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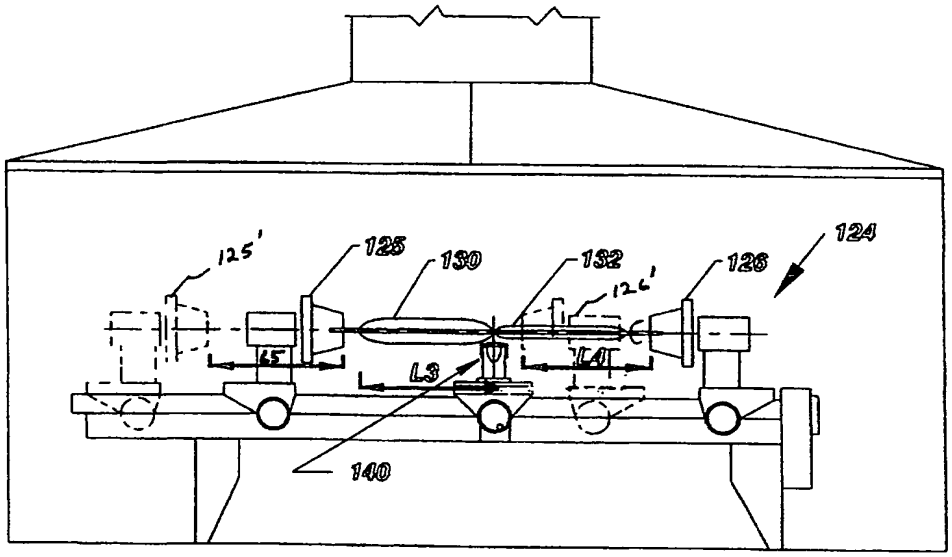

(57) Abstract

Glass soot (130) is deposited on a glass rod by a burner (140). The body is stretched (126). More depositing and stretching is effected. The final body is then drawn into a fiber.

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METHOD OF MAKING AN OPTICAL FIBER PREFORM

BACKGROUND OF THE INVENTION

The present invention relates to methods for making optical fiber preform of both single mode and multimode design using a plasma outside vapor deposition process.

The prior art teaches various approaches for fabricating silica glass starter tubes, and for making optical fiber preforms. Starter tubes can be formed by heating silica and extruding it through an aperture. Both starter tubes and optical fiber preforms can be made by depositing doped or undoped silica onto a target using one of several techniques such as modified chemical vapor deposition (MCVD), vapor axial deposition (VAD), outside vapor deposition (OVD). Each of these methods starts with providing a rotating target, typically shaped in the form of a tube or a solid rod, and formed from glass, ceramic or one of several other materials. In certain cases, the rod or tube becomes an integral part of the preform but, in other cases, the rod will be removed. A heat source, such as a gas burner or a plasma source is positioned beneath, above or laterally, across the rotating target. The heat source will provide the required energy for the glass-forming reactions to form glass particles. Depending upon the nature of the process, these deposited glass particles are ready for the next processing, drying and sintering steps such as VAD or OVD processes. If it is an MCVD process, these particles will be fused into vitreous quartz by the same heat source.

When the target is mounted horizontally, the heat source travels along the length of the target to ensure uniform deposition. If the target is a tube, the glass forming particles and materials may be deposited either on the inside surface of the tube, in which case the

outer diameter remains constant, or on the outside of the tube, in which case the outer diameter grows.

When the target is mounted vertically, it rotates around its vertical axis, and with burners located either vertically above or laterally across, grows in both radial and axial directions. This results in a substantially cylindrical product whose diameter and length increase as deposition continues.

UPS 3,737,292 to Keck et al. discloses a method of forming optical fibers. Multiple layers with predetermined index of refraction are formed by flame hydrolysis and deposited on the outside wall of a starting rod or member. After these layers of glass are coated on the rod the resulting hollow cylinder is heated and collapsed to form fibers.

USP 4,224,046 to Izawa et al. teaches a method for manufacturing an optical fiber preform. Two gaseous glass forming materials, oxygen, hydrogen and argon are jetted upwards in a burner towards a vertically mounted, rotating cylindrical start member. Soot-like glass particles are formed by flame hydrolysis and deposited on the lower end of the start member. The start member is gradually withdrawn upwards to maintain a constant spacing between the its growing end and the burner. Upon completion of the deposition, the resulting soot-like glass preform is then dried and sintered to form a transparent glass preform.

UPS 4,217,027 to MacChesney et al. teaches the fabrication of preforms by what is usually referred to as the Modified Chemical Vapor Deposition (MCVD) process. In this process, a vapor stream consisting of chlorides or hydrides of silicon and germanium with oxygen is directed to the inside of a glass tube. The chemical reactions among these chemicals, which are preferentially induced by a traversing hot zone, will under proper conditions result in the formation of glass on the inner wall of the tube. The particular matter deposited on the tube is fused with each passage of the hot zone.

USP 4,412,853 to Partus discloses an MCVD process to form an optical fiber preform starter tube. The process starts with a horizontally mounted, rotating tubular target formed from glass and having a preselected composition and optical characteristics. A vapor stream is fed through the tubular target as a heat source positioned beneath the tubular target, traverses along the latter's length. This causes reaction products of the vapor stream to be deposited on, and fuse to, the interior surface of the tubular target. The deposited material has the same index of refraction as the tubular target, but a different composition. This reference also suggests that one may achieve the same effect by an outside vapor-phase oxidation process or an outside vapor-phase axial deposition process, but does not explicitly teach how this can be done.

USP 4,741,747 to Geittner et al. is directed to the Plasma Chemical Vapor Deposition (PCVD) method of fabricating optical fibers. In this PCVD method glass layers are deposited on the inner wall of a glass tube by heating the tube to a temperature between 1100° and 1300° C, before passing the reactive gas mixture at a pressure between 1 and 30 hPa, and moving a plasma back and forth inside the glass tube. After the glass layers are deposited, this glass tube is collapsed to produce a solid preform. Optical fibers can be drawn from this preform.

USP 5,522,007 to Drouart et al. teaches the use of plasma deposition to build up an optical fiber preform having high hydroxyl ion concentration. In this reference, hydroxyl ions are deliberately entrained in a plasma generating gas by passing the gas through a water tank before it is introduced into one end of a plasma torch having an induction coil. The plasma torch projects molten silica particles mixed with hydroxyl ions onto a rotating substrate preform. This results in a preform having an average hydroxyl ion concentration lying in the range to 50-100 ppm deposited onto the

target preform. According to Drouart et al., this technique results in optical fibers having an attenuation of 0.32 dB/km and 0.195 db/km at 1310 nm and 1550 nm, respectively.

In addition to requiring multiple processing steps to fabricate preforms, some other disadvantages of the above processes are that:

1. the MCVD and PCVD processes are slower processes because of their low deposition rate;

2. the preform size is limited by the size of the deposition tube for MCVD and PCVD process; and

3. the OVD and VAD processes are based on flame hydrolysis which generates excessive amounts of water and requires additional drying and sintering steps to produce high quality optical fiber preforms.

SUMMARY OF THE INVENTION

It is an object of the invention to provide a method for producing an optical fiber preform having low hydroxyl content at low cost by reducing the number of steps entailed in its manufacture, while increasing the size of a preform and increasing the rate of deposition. This and other objects are achieved by the present inventive method for forming an optical fiber preform.

In one aspect of the present invention, a plasma source is placed in proximity to a starter rod formed from a primary material. The starter rod is held horizontally at both ends and is arranged to rotate about its longitudinal axis. The plasma source is used to deposit silica doped with a known first doping concentration. The doped silica is deposited along the length of the starter rod until the latter grows to a desired diameter. The complex comprising the starter rod and the doped silica is then drawn down and a thinned section is extracted for use as a secondary rod. The secondary rod has a center formed from the primary material, and an outer layer formed from the doped silica. Additional silica, having the same doping

concentration, is deposited atop this secondary rod until it, too, reaches a desired diameter, and then is drawn down and a section extracted. The steps of depositing drawing down, extracting and depositing may be repeated a number of times. The result of this activity is a doped silica rod having a center formed from the primary material with a first diameter, and an annular layer formed from the doped silica with a second outer diameter.

The doped silica rod is subject to further processing. Specifically, the plasma source is used to deposit an outer layer of doped silica atop the doped silica rod and the resulting structure may then be drawn down and a thinned section extracted, as before. The dopant used in forming the outer layer may be selected to either increase, or decrease, the index of refraction of the silica.

If the dopant concentration is varied as the outer layer is being deposited, the outer layer is a graded layer. In such case, typically, the dopant concentration is varied from a maximum, beginning concentration level when the outer layer is first being deposited, to a minimum, end concentration level when deposition of the outer layer is almost complete.

If the dopant concentration is not varied as the outer layer is being deposited, the outer layer is a stepped layer. In such case, typically, a second dopant concentration, different from the first dopant concentration, is used throughout the deposition of the outer layer.

In yet another aspect of the present invention, the complex comprising the doped silica rod and the outer layer is subjected to further processing. The plasma source is used to deposit a cladding layer atop the outer layer. If the outer layer was graded, the cladding layer may be formed from silica doped with the same dopant and same minimum, end concentration level. Alternatively, the cladding layer can be formed from pure silica, or

even silica doped with some other dopant and at a third dopant concentration. If desired, the cladding layer may also have a graded doping.

In yet another aspect of the present invention, the complex comprising the doped silica rod, the outer layer and the cladding layer, is provided with a jacket. The jacket can be added by either further plasma deposition, or, alternatively, by providing a jacketing material over this complex and then applying heat to collapse the jacketing material into a finished preform.

During plasma deposition, a dry plasma gas having a low hydroxyl concentration is used to form the plasma. A dry quartz source gas comprising $SiCl_4$, or other similar source gases having low hydroxyl concentration, and a dopant source gas such as $GeCl_4$, which is sometimes co-doped with $POCl_3$ or PCl_5 are introduced in proximity to the plasma. This causes the material to be converted to silica (SiO_2), or silica doped with germanium oxide (GeO_2) and or phosphorous pentoxide (P_2O_5) and deposited onto the target and fused into vitreous quartz in one simple step.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other features, aspects, and advantages of the present invention can be seen in the drawings in which:

Fig. 1 shows an apparatus used to perform plasma deposition;

Fig. 2 shows a partial side view of a plasmatron used in the apparatus of Fig. 1;

Fig. 3 shows a top view of a plasmatron similar to that shown in Fig. 2;

Fig. 4 shows a flow pattern of the plasma within the plasmatron of Fig. 3;

Fig. 5 shows an optical fiber preform made in accordance with the method of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Fig. 1 shows an apparatus 20 used for plasma outside vapor deposition. The apparatus comprises a chamber 22 which is sealed so as to prevent impurities from being introduced into the final product.

Within the chamber 22 is a lathe 24, such as that available from Heathway Ltd. or Litton Engineering Lab. The lathe 24 has a headstock 25 and a tailstock 26. The headstock 25 and the tailstock 26 are provided with a pair of opposing rotating spindle chucks 28 which hold the ends of an elongated target 30 having a substantially cylindrical outer wall. The spindle chucks 28 rotate target 30, as indicated by arrow A1. A movable carriage 32 movably mounted to the lathe 24 is arranged to travel in either direction along the target, as indicated by double headed arrow A2.

A plasma source, shown generally as 40, is supported by carriage 32. Carriage 32 thus moves plasma source 40 along the length of the target 30. This results in the deposition of material on top of the target 30 to form an optical fiber preform. The spindle chucks 28 rotate the target 30 to ensure that material is uniformly deposited by the plasma source 40 around the target so as to form a tubular member 34 having nearly perfectly cylindrical outer walls.

In the preferred embodiment, the plasma source 40 positioned on the carriage 32 moves in both directions along a substantial portion of the length of the target 30. This allows the plasma source 40 to travel along this portion of the target 30 and deposit materials therealong.

Instead of moving the plasma source 40 along the length of the target, the target 30 may be moved while the plasma source 40 remains stationary. This can be realized by having the headstock 25 and the tailstock 26 of the lathe move the target in a reciprocating fashion so that all relevant portions of the target are brought directly above the plasma source 40.

As another alternative, a plurality of plasma sources may be spaced apart along the length of the target. This allows for reduced movement of either the headstock 25 and tailstock 26 of the lathe 24, or the carriage 32 to which the plasma sources are attached, depending on which of the two is configured to move. In the extreme case where a great number of plasma sources are provided all along the length of the target, no movement of either the carriage 32 or the headstock 25 and tailstock 26 of the lathe 24 is needed.

In the preferred embodiment, the plasma source 40 is a plasmatron torch having a dry plasma gas introduced into it through a first gas line 42 and a source gas introduced into it through a second gas line 44.

The plasma gas is substantially comprised of nitrogen and oxygen in an appropriate, predetermined proportion. Air may serve as the plasma gas. In such case, filtered air first passes through a first dryer 46 to remove moisture before entering the first gas line 42. This ensures that the hydroxyl concentration of the plasma gas is low, on the order of 2.0 ppm, or less. The total volume of gas being delivered will be regulated by a mass flow controller (MFC) 80 or by a flowmeter, as an alternative.

The source gas comprises a source chemical such as $SiCl_4$, and at least one carrier gas, such as oxygen O_2 or nitrogen N_2. The carrier gases enter the second dryer 48 to remove moisture. This ensures that the hydroxyl concentration of the source gas is also very low, on the order of 0.5 ppm. After the carrier gases are dried, they proceed to a MFC 81 before entering a bubbler 50 to pick up the source chemical. Depending upon the characteristics of the MFC, it is also possible to use it downstream of the bubbler. The gas stream comprising carrier gases laden with the source chemical then proceeds to the second gas line 44. Optionally, by opening valve 52, a dopant gas may be introduced into the gas stream before it reaches the plasmatron torch.

In the preferred embodiment, the source chemical is $SiCl_4$. This chemical is chosen for its reactive properties in a plasma. Specifically, the $SiCl_4$ serves as a source of Si to form SiO_2 which is deposited on the target 30. The dopant can be a fluorine dopant gas in the form of SiF_4 or SiF_6. Fluorine dopants will lower the index of refraction and also change the viscosity of the quartz. In addition, fluorine dopants result in increased design flexibility for optical fiber preforms. As is well known, however, if one wishes to increase the index of refraction, GeO_2 or other equivalent substance may be used as the dopant.

In the preferred embodiment, the source chemical for GeO_2 is $GeCl_4$. This chemical is chosen for its purity because of its having similar physical and chemical properties $SiCl_4$. The delivery of the $GeCl_4$ will be similar to $SiCl_4$. The carrier gas from the dryer 48, can be split to another branch where it will be regulated by a MFC 82, before proceeding to a bubbler 83 to pick up the source chemical $GeCl_4$. Similar to the control of chemical $SiCl_4$, the MFC can also be located downstream of the bubbler. This gas stream can feed into the gas line 44 and form a mixture before entering the plasmatron torch. It is also possible to directly introduce the $GeCl_4$ gas stream by a separate line 84 to the plasmatron torch. One advantage of using the separated delivery lines is to minimize the competing chemical reactions between $GeCl_4$ and $SiCl_4$. Other source chemicals that can be used for doping instead of germanium oxide (GeO_2) or co-doping with germanium oxide are materials such as $POCl_3$, PCl_5, and other similar index increasing dopants such as Aluminum and Titanium containing chemicals.

Fig. 2 shows a cutaway side view of the plasmatron torch 40 positioned below the target 30. The plasmatron torch 40 comprises a substantially tubular torch housing 50 formed from quartz. The housing has a diameter of 60 mm and a height of 220 mm. However, diameters ranging

from 40-80 mm and heights between 180-400 mm may also be used.

A copper induction coil 52 is provided around the upper portion of the housing 50. The coil 52 comprises a plurality of windings 54 having a diameter of approximately 72 mm and spaced apart from each other by 6 mm. A gap between the housing and the coil can be between 2-10 mm. The uppermost portion of the coil 52, as indicated by uppermost winding 54', is separated from the outer surface of the tubular member 34 by a spacing designated by L, which is on the order of 30-55 mm.

As the quartz glass is deposited, its outer diameter increases. However, the spacing L is maintained by adjusting the height of a support stand 56 on which the plasma torch 40 is placed. Support stand 56, in turn, is mounted to carriage 32, and moves laterally therewith. Initially, the support stand 56 is set at a predetermined height, and this height is reduced as the diameter of the deposited material increases during deposition. This maintains a predetermined distance between the plasma torch 40 and the deposited material. An optical or other sensor mounted on the carriage 32 and connected to a controller may be used to gauge the distance of the radially growing tubular member 34 from the carriage, and adjust the height of the support stand 56, accordingly.

On either side of the uppermost portion of the housing 50 is a plasma stabilizer bar 58. Each stabilizer bar is formed from quartz and comprises a U-shaped gutter extending laterally from the rim of the housing 50. The stabilizer bars 58 have a diameter of 60 mm and extend 20 mm on diametrically opposite sides of the housing rim, although diameters in the range of 40-80 mm and lengths of 15-40 mm may also be used. When the plasmatron torch 40 is in use, the stabilizer bars 58 are aligned parallely to the target. This arrangement helps spread the reactive source chemicals being deposited onto the growing tubular member 34.

A pair of injection ports 60 connect the second gas line 44 carrying the source chemicals to the plasmatron torch 40. The injection ports 60 enter the housing at substantially the same height along the housing 50, at a point between the uppermost windings 54' of the coil 52 and the stabilizer bars 58. The injection ports comprise quartz tubing having a diameter of 5 mm, although tubing diameters on the order of 3-10 mm may be used with the plasmatron torch 40 of the present invention. In the preferred embodiment, a pair of injection ports 60 enter the housing 50 at the same height and are positioned diametrically across from each other. Instead of just two such ports, however, three or even more ports, symmetrically arranged, may be provided. In Fig. 2, the two injection ports 60 are shown to be directly beneath the stabilizer bars. This, however, is not an absolute necessity, and the injection ports 60 may be angularly offset from the stabilizer bars 58, in a top view of the plasmatron torch, as shown in Fig. 3.

A pair of plasma gas inlets 62 connect the first gas line 42 carrying the plasma gases to the plasmatron torch 40. The plasma gas inlets 62 enter the housing at substantially the same height, proximate to the base of the housing. These inlets 62 comprise stainless steel tubing having a diameter of 5 mm, although a range of diameters may suffice for this purpose.

The plasmatron torch 40 is also provided with a coolant inlet 64 and outlet 66. During use, a coolant, such as water, passes through the inlet 64, circulates within the outer wall of the housing 50, and exits through the outlet 66. The coolant inlet and outlet are formed from stainless steel and have a diameter of 5 mm. As with the plasma gas inlet and the injection port, this diameter may also vary.

The plasma gas inlets 62, the coolant inlet 64 and the coolant outlet 66 are all formed in a stainless steel chamber 68. The chamber 68 is a stainless steel square block 80 mm on a side, and having a height of

approximately 40 mm. The chamber 68 is mounted onto the support stand 56 which, in turn, is mounted on the carriage 32 for movement along the target 30.

A high frequency generator (not shown) is electrically connected to the coil 52, powering the latter with a variable power output up to 80 kW at a frequency of approximately 5.0 MHz. In the preferred embodiment, the generator is Model No. T-80-3MC from Lepel Corporation. This generator is driven with a 60 Hz, 3-phase 460 V power supply to energize the plasmatron torch 40. As an alternative, a Model No. IG 60/5000 generator is available from Fritz Huttinger Electronic GmbH of Germany.

Fig. 4 depicts the plasma jet 70 formed within the plasmatron torch 40 when the dry plasma gas is fed through the inlets 62 and converted into a plasma. The plasma jet 70 is substantially symmetric about the torch's longitudinal axis A'. The position of the injection ports 60 is such that the source chemicals are introduced into the plasma just above a point V where the vertical velocity of said plasma is zero. This provides the needed structure of hydrodynamic and thermal flow of the source chemical jet into the border layers to realize efficient deposition onto the growing tubular member 34. And while the preferred embodiment has the injection ports entering laterally into the housing, this is not an absolute requirement. Instead, the source gases may introduced into the center of the plasma jet 70 by a water cooled probe extending along the longitudinal axis A' of the plasmatron torch 40.

Fig. 5 illustrates a well-known procedure which can performed with a lathe 124, such as Model No. PFH842XXLS Precision Quartz and Glass Working Lathe, manufactured by Heathway. The headstock 125 and tailstock 126 of the lathe 124 can move longitudinally relative to one another. This allows for easy loading and unloading of a finished workpiece 130 of length L3 which has been deposited atop an initial target. More significantly, it

also allows one to draw down a portion of a workpiece into a secondary rod of a reduced diameter comparable to that of the original target. This is accomplished by keeping the headstock 125 stationary and moving the tailstock 126 away from the headstock 125 while the plasma source 140 is moved in a direction opposite to that of tailstock 126. Alternatively, this can also be accomplished by placing a plasma source 140, or other heat source, at one end of the workpiece 130 to soften it. Then the headstock 125 and tailstock 126 are moved in the same direction, but with different speeds by distances L5, L4, respectively, to the positions shown in phantom 125', 126'. The result is a thin, secondary rod 132, which can (but need not) have the same diameter as the original target. As is known to those skilled in the art, the secondary rod has the same cross-sectional composition as the workpiece from which it is derived, as so has a center whose consistency along is substantially similar to that of the original target, and outer layer substantially similar to the materials deposited atop the target during the formation of the workpiece.

The lathe 124 allows the headstock 125 and tailstock 126 to be moved far enough longitudinally to stretch the secondary rod to a distance L4, which is substantially the same as the length L3 of the workpiece from which it is derived. The secondary rod 132 may be cut from the workpiece, mounted on the lathe 124 in place of the workpiece 130, and used as a target for subsequent deposition with the plasma source 140. Thus, the original, or first-generation, target is used to create a first-generation workpiece, from which a secondary rod can be drawn to be used as a second-generation target. Deposition atop this second-generation target can will thereby form a second-generation workpiece, and so on. This iterative process of plasma deposition on a target to form a workpiece, stretching one end of the workpiece to form a reduced-diameter rod, and using this reduced-

diameter rod as a subsequent target for further deposition can be repeated an arbitrary number of times.

If the material being deposited atop the target is unchanged through the iterations, the result of N iterative steps is an N-th generation rod having a very small center which is substantially identical in composition to the original target, and an annular layer reflective of the materials deposited atop the target. For instance, if the original target has a diameter D1 and the finished workpiece has a diameter D2 = M x D1, then the proportion of the original target material in the first-generation workpiece is approximately $1/M^2$. If a second-generation target of diameter D1 is drawn from this workpiece and material sufficient to form a second-generation workpiece of diameter D2 is deposited thereon, the proportion of the original target material in the second generation workpiece is approximately $1/M^4$. Thus, it can be seen that one may readily form a workpiece having a predetermined proportion of the original target material therein by controlling M during deposition, along with the total number of iterations.

A method for forming a multimode optical fiber preform using the aforementioned iterative technique will now be described. In order to provide a more detailed explanation, some dimensions are given. However, it must be noted that in the actual process, many different value are possible.

The method begins by providing a first generation target, horizontally mounted on a lathe, such as that shown in Fig. 5. The target is preferably formed from pure silica, in which case it may be purchased from a commercial vendor, such as Product no. F300, available from Heraeus Amersil of Georgia. Alternatively, the first-generation target may be an Nth-generation doped silica rod formed using the current process. In the preferred embodiment, the first-generation target has a length of one meter and a diameter D1 = 6 mm.

Silica doped with GeO_2 is deposited atop the first-generation target using the plasma source described above. The dopant concentration for the GeO_2 depends on the desired numerical aperture (NA) of the multimode optical fiber being produced. For instance, to form a fiber with a NA of 0.2, the maximum GeO_2 dopant concentration is approximately 10%. And to form a fiber with a NA of 0.275, the maximum GeO_2 dopant concentration will be approximately 18%.

The dopant concentration may be held at the same level during deposition, in which case a stepped layer, is formed. Alternatively, the dopant concentration may be gradually varied to form a graded layer. This is done by automatically controlling, by means of a microprocessor or like, an adjustable flow meter through which the dopant is introduced. It should be noted that stepped and graded layers may succeed one another in subsequent generations of workpieces, and that layers having different, constant doping concentrations may succeed one another, as well. Thus, a graded layer may be deposited on the first-generation target, and a stepped layer may be deposited atop the second-generation target formed after drawing down the first-generation workpiece. Similarly, one may deposit a stepped layer atop a graded layer, which has been deposited atop an original first-generation target. Also, a first stepped layer, having a first dopant concentration, may be deposited atop a target, and a second stepped layer, having a second dopant concentration, deposited atop the next generation target. Additional layers, either graded or stepped, may be deposited atop any of the above structure.

In the preferred embodiment, silica doped with 18 % GeO_2 is deposited as a stepped layer atop the 6 mm diameter first generation target until a workpiece having a length of one meter and a diameter of D2 = 48 mm is formed (i.e., M = 8). This resulting first-generation workpiece has approximately 64 times the cross-sectional

area of the original first-generation target. The first-generation workpiece is then drawn down into 64 first-generation doped silica rods, each having a length of one meter and a diameter of 6 mm. Each of these doped silica rods may then be used as a second-generation target.

The second generation target is placed in a lathe and a second deposition layer is applied to form a second-generation workpiece having a 48 mm diameter. This second deposition is carried out with the same, constant dopant concentration as the first deposition. Maintaining the dopant concentration at the same level throughout the deposition process results in a first-generation doped silica rod with a center formed from the original target material and an annular layer which has substantially the same composition therethrough. This ensures that the optical properties of the second layer is substantially the same as that of the first layer which was deposited on the original target. The second-generation workpiece is then drawn down into 144 second-generation doped silica rods, each having a length of one meter and a diameter of 4 mm. Each of these may be used as a third-generation target. It should be noted here that the iterative process may continue with the deposition of additional layers having the same dopant concentration. At some point, however, a workpiece with a desired proportion of original target material will be formed, after which no further iterations are needed. Indeed, this may even be reached after the first generation workpiece is formed.

In the preferred embodiment, a graded deposition layer having an outer diameter of approximately 80 mm is deposited atop the 4 mm diameter third-generation target. The dopant concentration starts out at a maximum value of 18% GeO_2 closest to the outer surface of the third-generation target, and is gradually reduced to a minimum value approximately 0.1% GeO_2 at its outermost portion, where the diameter is about 80 mm. This results in a third-generation workpiece having a center formed from

the original target, two layers having substantially the same optical properties and fairly indistinguishable from one another, and a third, graded layer.

In the preferred embodiment, the 80 mm diameter third-generation workpiece is subject to additional processing to form a primary optical fiber preform. Specifically, a cladding, or barrier, layer is deposited atop the third-generation workpiece. The thickness of the cladding layer depends on the type of finished optical fiber preform to be made. For a 62.5/125 fiber preform, the finished primary preform will have a final diameter of about 93 mm. For a 50/125 fiber preform, the finished primary preform will have a final diameter of about 96 mm. The cladding layer is formed by depositing silica doped at the same concentration of GeO_2 as the minimum doping concentration level used to form the third layer, i.e., 10% GeO_2. This results in a structure having the original target material at the center, a constantly doped pair of second layers having the same optical properties, a graded layer having a dopant concentration varying from a maximum value to a minimum value, and a cladding layer comprising silica doped at the minimum value.

Once the cladding layer is applied, the finished primary preform must be stretched to form the final preforms. From a single, 1 meter long 62.5/125 preform having a diameter of 93 mm diameter, one can obtain eight, one-meter long preform pieces, each having an outer diameter of 32 mm. And from a single, 1 meter long 50/125 preform having a diameter of 96 mm diameter, one can obtain twelve, one-meter long pieces, each having an outer diameter of 27 mm.

A jacketing layer may be applied atop the cladding layer of these preform pieces. The jacketing layer preferably has the same index of refraction as pure silica. The jacket may be applied by plasma outside vapor deposition using pure silica. Alternatively, a tube or sheet of pure silica, having an appropriate

diameter or width, may be provided around a preform piece, and heat applied to fuse the jacket onto the preform piece to form the final optical fiber preform. In the preferred embodiment, the final optical preform has an outer diameter of about 56 mm. This final preform may then be drawn into approximately 200 Km of fiber having a diameter of 125 μm.

Although, for best performance, a cladding and then a jacketing layer is applied, it should be noted that one may dispose of the cladding step and directly apply a jacketing tube to the third-generation workpiece, once it has been stretched.

A similar method for making single mode optical fiber preform can be achieved by using the following procedure. The starting target can be a pure silica rod that can be either a F300 rod purchased from Heraeus or a pure silica Nth-generation rod fabricated in house. Multiple fluorine doped silica layers with constant concentration are deposited on the target until it reaches a desired diameter. Single mode optical fibers can be drawn from this preform. There are many different glass index modifiers such as F, GeO_2, P_2O_5, TiO_2, Al_2O_3, etc., and in the proper combination, they can be used to make the doped core and/or doped cladding. In the preferred embodiment, the target is a Nth-generation GeO_2 doped rod with pure silica or doped silica cladding layers deposited on it. The preform is completed when the desired diameter is reached.

While the present invention has been disclosed with reference to certain preferred embodiments, these should not be considered to limit the present invention. One skilled in the art will readily recognize that variations of these embodiments are possible, each falling within the scope of the invention, as set forth in the claims below.

CLAIMS

What is claimed is:

1. A method for making an optical fiber preform comprising the steps of:

(a) providing a target rod formed from a first material;

(b) depositing atop said target rod, a first silica layer doped with a first dopant provided at a first concentration, said first silica layer being deposited to a predetermined first thickness;

(c) drawing down said target rod with said first silica layer deposited thereon to a predetermined first diameter, thereby forming a doped silica rod;

(d) repeating steps (b) and (c)

(d1) for a predetermined number of times, or

(d2) until said first material comprises a predetermined proportion of said doped silica rod;

(e) depositing atop said doped silica rod, a second layer comprising silica doped with a second dopant provided at a second concentration, said second silica layer being deposited to a predetermined second thickness to thereby form an intermediate structure;

(f) depositing a third layer atop said intermediate structure, said third layer being deposited to a predetermined third thickness to thereby form a preform structure.

2. The method of claim 1, comprising the additional step of:

(g) applying a jacketing layer atop said preform structure, said jacketing layer consisting essentially of pure silica and being applied to a predetermined fourth thickness.

3. The method of claim 2, comprising the additional step of:

drawing down said preform structure to a predetermined third diameter after step (f) and prior to step (g).

4. The method of claim 1, wherein the first material is one from the group consisting of silica and silica doped with a dopant.

5. The method of claim 4, wherein the dopant is an index modifying material which is one from the group consisting of F, GeO_2, P_2O5, TiO_2 and Al_2O_3.

6. The method of claim 1, wherein, in step (e), said second concentration differs from said first concentration, said method comprises the additional step of:

maintaining the second concentration at a constant value as the second silica layer is being deposited, thereby forming a step index profile in the indices of the refraction of the doped silica rod and the second silica layer.

7. The method of claim 1, comprising the step of varying the second concentration as the second silica layer is deposited.

8. The method of claim 7, wherein
the second dopant is fluorine, and
said second concentration is varied from a minimum value when said second silica layer is first being deposited, to a maximum value when deposition of said second silica layer is nearing completion.

9. The method of claim 7, wherein said second concentration is varied from a maximum value when said second silica layer is first being deposited, to a minimum value when deposition of said second silica is nearing completion.

10. The method of claim 9, wherein said maximum value of said second concentration is substantially the same as said first concentration.

11. The method of claim 9, wherein the third layer deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with said second dopant at said minimum value.

12. The method of claim 9, wherein the third layer deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with fluorine.

13. The method of claim 1, wherein the third layer deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with fluorine.

14. The method of claim 9, comprising the additional step of:

(g) applying a jacketing layer atop said preform structure, said jacketing layer consisting essentially of pure silica and being applied to a predetermined fourth thickness.

15. The method of claim 14, comprising the additional step of:

drawing down said preform structure to a predetermined third diameter after step (f) and prior to step (g).

16. The method of claim 1, wherein at least one of said depositing steps (b), (e) and (f) is performed by plasma outside vapor deposition, which comprises the steps of:

providing a high-frequency plasmatron comprising a coil, said plasmatron being selectively positionable along a length of said target with a spacing of 30-55 mm separating the target from said coil;

introducing a plasma gas having a hydroxyl content of less than 2 ppm into the plasmatron to form a plasma;

injecting a source gas comprising at least $SiCl_4$ and a dopant into a region in communication with said plasma, said source gas having a hydroxyl content of less than 0.5 ppm; and

depositing at least one reaction product of said plasma and said source gas onto the target while maintaining said spacing between the target and the coil.

17. The method of claim 16, wherein the source gas is introduced just above a point in the plasmatron at which the vertical velocity of the plasma is zero.

18. The method of claim 16, wherein said coil comprises a plurality of windings, and the target is separated from a winding closest to the target by said spacing.

19. The method of claim 17, comprising the additional step of drying the plasma gas before it is introduced into the plasmatron.

20. The method of claim 1, wherein the first and second dopants and the first and second concentrations, are the same.

AMENDED CLAIMS

[received by the International Bureau on 10 August 1999 (10.08.99) ;
original claims 1, 6, 16, 17, 18, 19 and 20 amended ;
remaining claims unchanged (4 pages)]

1. A method for making an optical fiber preform comprising steps of:

(a) providing a target rod formed from a first material;

(b) concurrently depositing and sintering on said target rod, by plasma torch, a first silica layer doped with a first dopant provided at a first concentration, said first silica layer being deposited and sintered to a predetermined first thickness;

(c) drawing down said target rod with said first silica layer deposited thereon to a predetermined first diameter, thereby forming a doped silica rod;

(d) repeating steps (b) and (c)

(d1) for a predetermined number of times, or

(d2) until said first material comprises a predetermined proportion of said doped silica rod;

(e) depositing on said doped silica rod a second layer comprising silica doped with a second dopant provided at a second concentration, said second silica layer being deposited to a predetermined second thickness to thereby form an intermediate structure;

(f) depositing a third layer on said intermediate structure, said third layer being deposited to a predetermined third thickness to thereby form a preform structure.

2. The method of claim 1, comprising the additional step of:

drawing down said preform structure to a predetermined third diameter after step (f) and prior to step (g).

4. The method of claim 1, wherein the first material is one from the group consisting of silica and silica doped with a dopant.

5. The method of claim 4, wherein the dopant is an index modifying material which is one from the group consisting of F, GeO_2, P_2O5, TiO_2 and Al_2O_3.

6. The method of claim 1, wherein, in step (e), said second concentration differs from said first concentration, and said method comprises the additional step of:

maintaining the second concentration at a constant value as the second silica layer is being deposited, thereby forming a step index profile in the indices of the refraction of the doped silica rod and the second silica layer.

7. The method of claim 1, comprising the step of varying the second concentration as the second silica layer is deposited.

8. The method of claim 7, wherein the second dopant is fluorine, and said second concentration is varied from a minimum value when said second silica layer is first being deposited, to a maximum value when deposition of said second silica layer is nearing completion.

9. The method of claim 7, wherein said second concentration is varied from a maximum value when said second silica layer is first being, deposited, to a minimum value when deposition of said second silica is nearing completion.

10. The method of claim 9, wherein said maximum value of said second-concentration is substantially the same as said first concentration.

11. The method of claim 9, wherein the third layer deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with said second dopant at said minimum value.

12. The method of claim 9, wherein the third layer

deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with fluorine.

13. The method of claim 1, wherein the third layer deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with fluorine.

14. The method of claim 9, comprising the additional step of:

(g) applying a jacketing layer on said preform structure, said jacketing layer consisting essentially of pure silica and being applied to a predetermined fourth thickness.

15. The method of claim 14, comprising the additional step of:

drawing down said preform structure to a predetermined third diameter after step (f) and prior to step (g).

16. The method of claim 1, wherein at least one of said steps (b), (e) and (f) is performed by plasma outside vapor deposition, which comprises steps of:

providing a high-frequency plasma torch comprising a coil having a plurality of windings around a coil axis, said plasma torch being selectively positionable along a length of said target with a spacing of 30-55 mm separating the target from said coil;

introducing a plasma gas having a hydroxyl content of less than 2 ppm into the plasma torch to form a plasma;

injecting a source gas comprising at least $SiCl_4$ and a dopant into a region in communication with said plasma, said source gas having a hydroxyl content of less than 0.5 ppm; and

depositing at least one reaction product of said plasma and said source gas onto the target while maintaining said spacing between the target and the coil.

17. The method of claim 16, wherein the source gas is introduced just above a point in the plasma torch at which the velocity of the plasma in the direction of said coil axis is zero.

18. The method of claim 16, wherein the target is separated from a winding closest to the target by said spacing.

19. The method of claim 17, comprising the additional step of drying the plasma gas before it is introduced into the plasma torch.

20. The method of claim 1, wherein the first and second dopants and the first and second concentrations are the same.

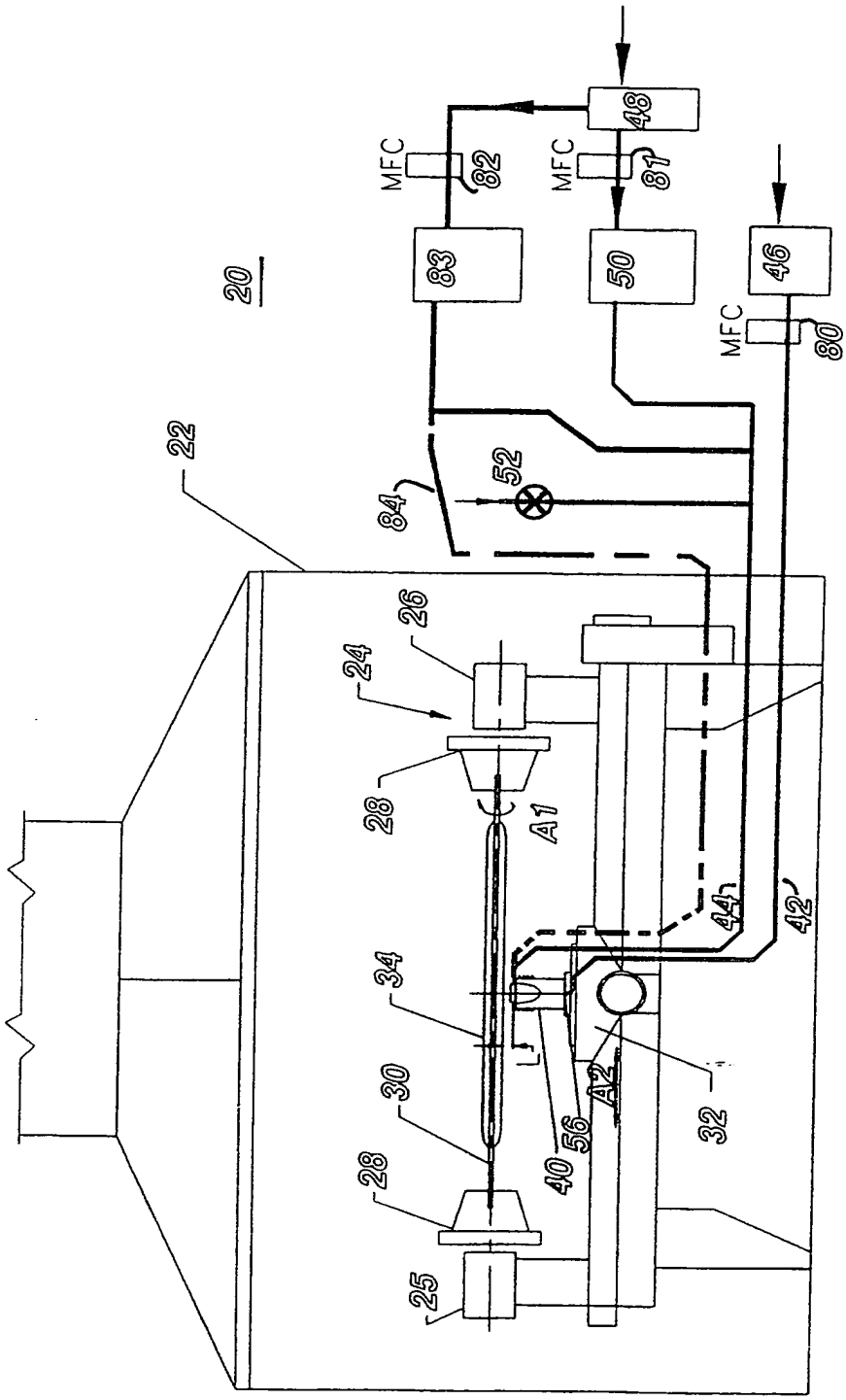
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Fig. 1

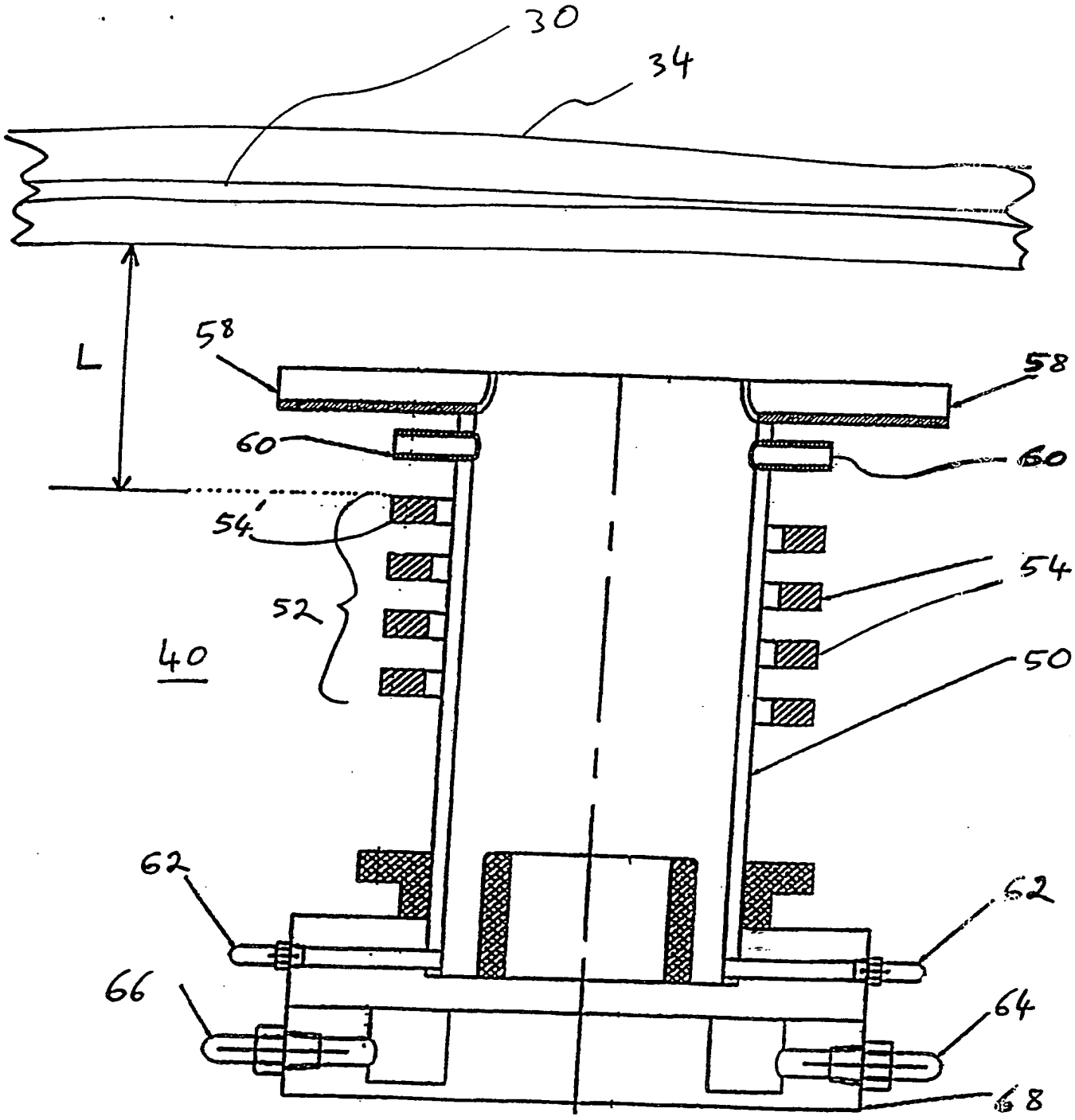

Fig. 2

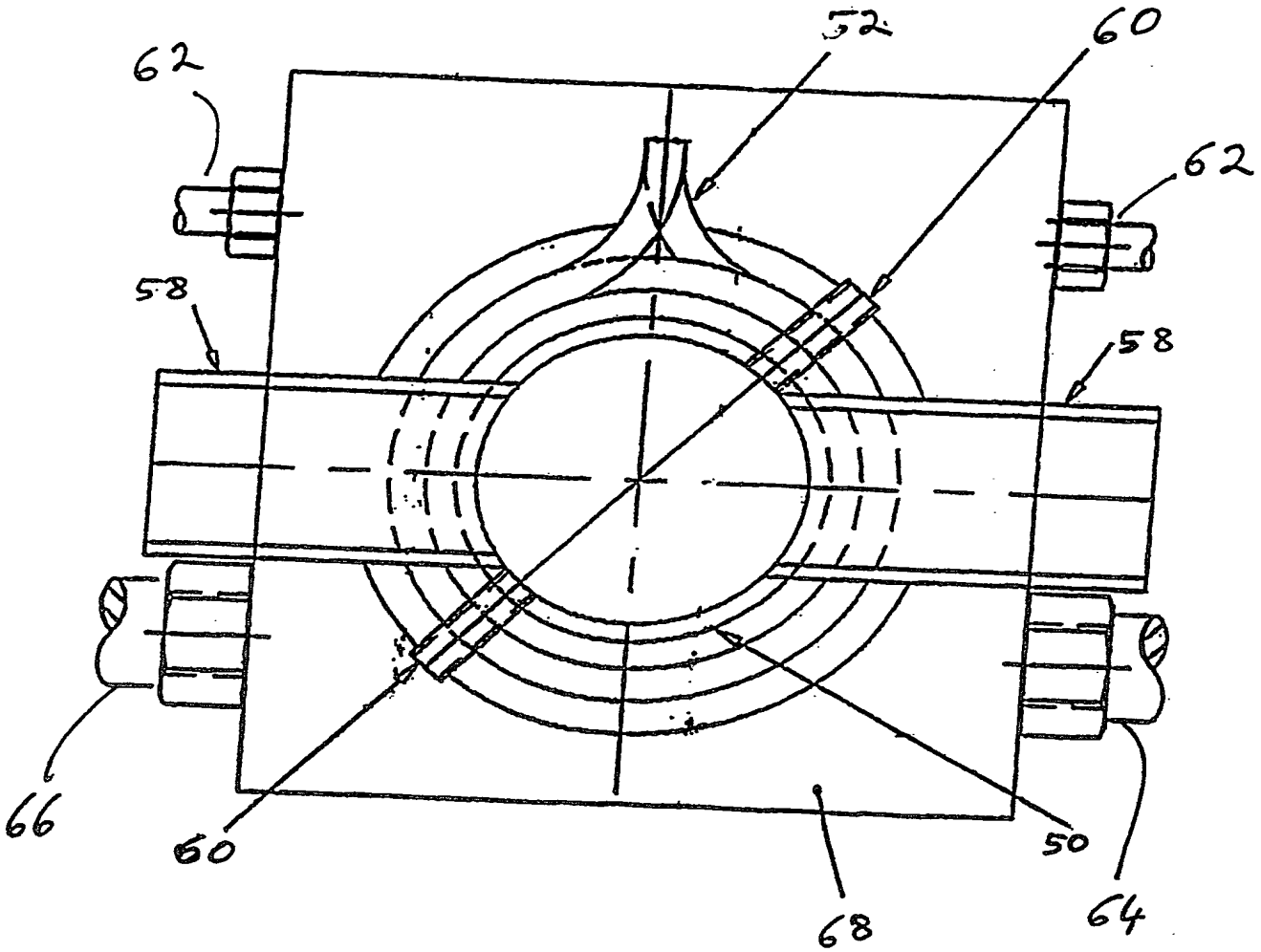

Fig. 3

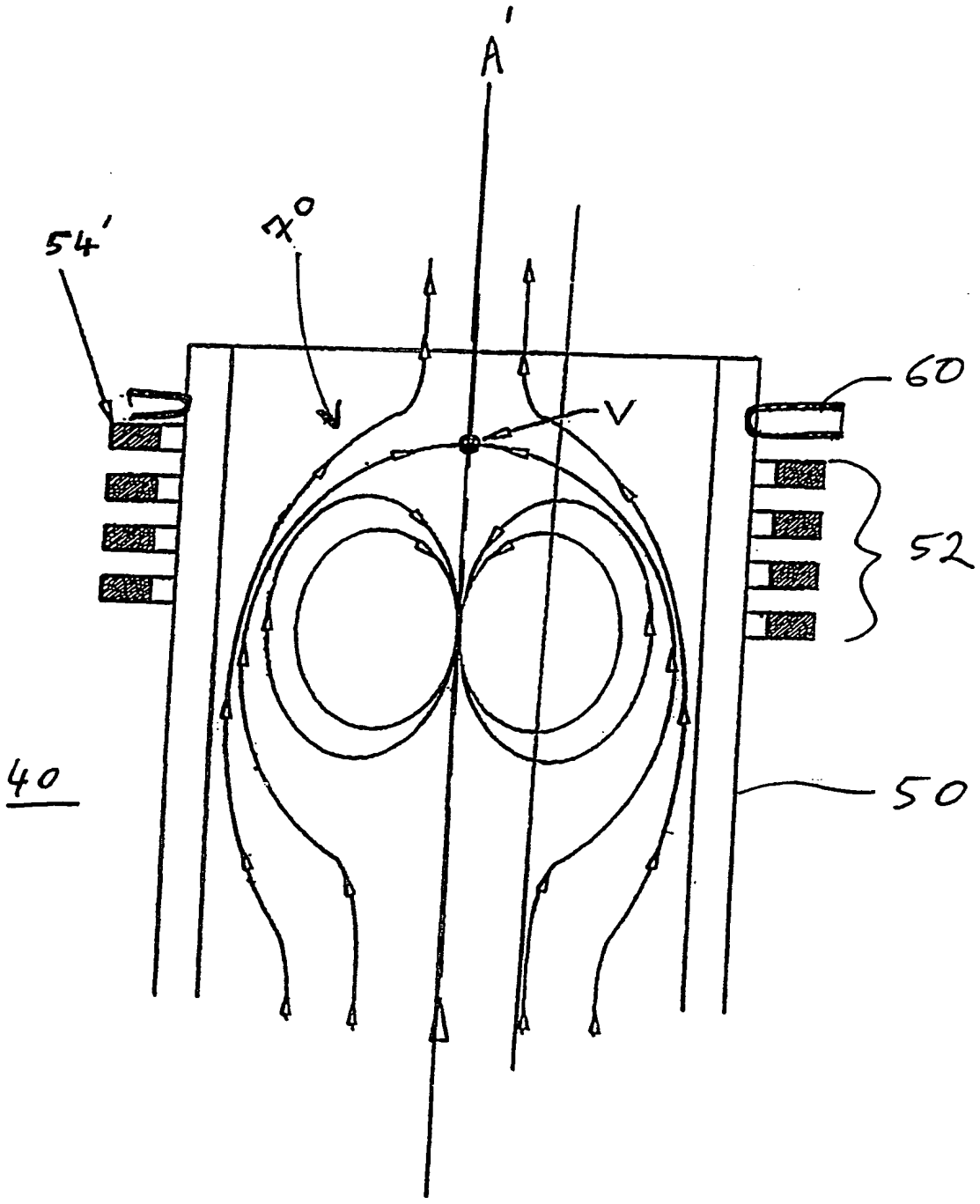

Fig. 4

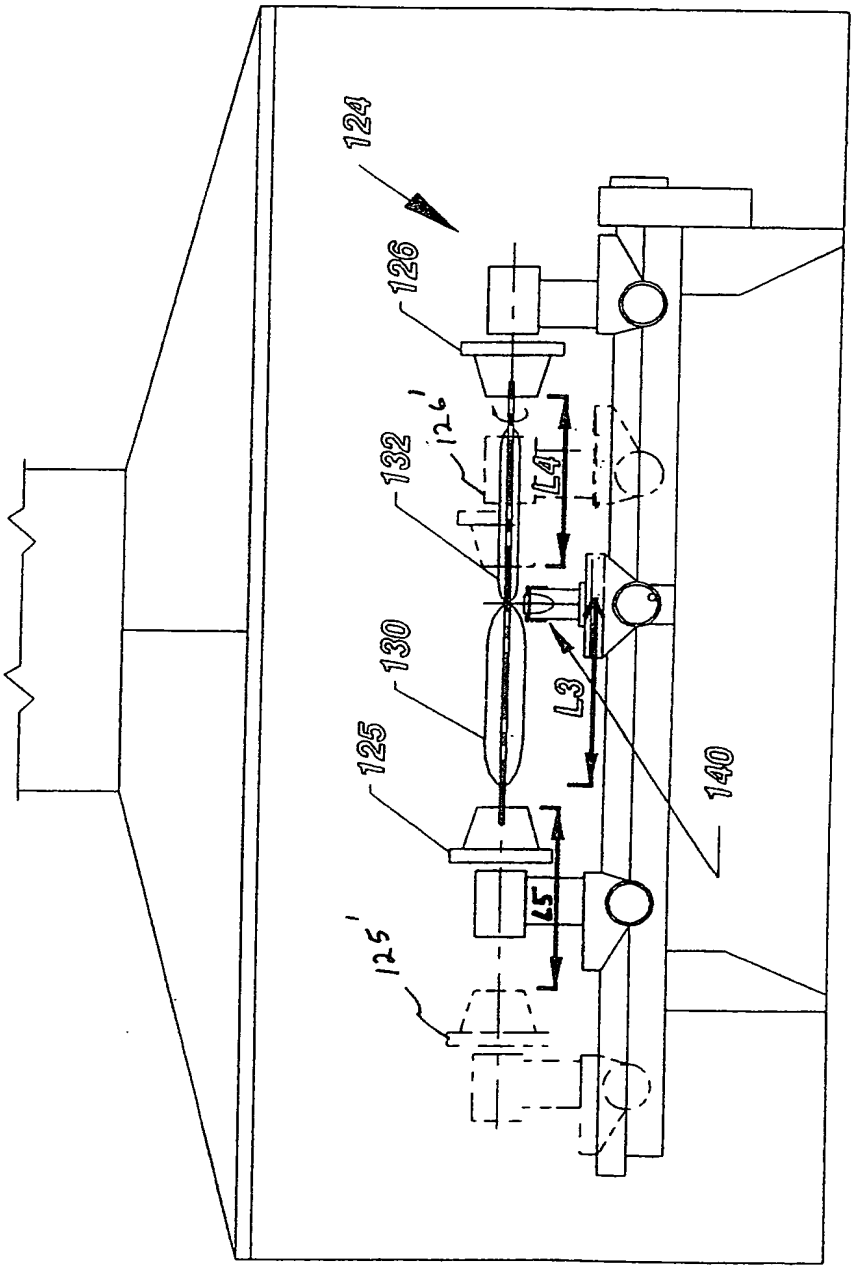

Fig.5

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US99/07872

A. CLASSIFICATION OF SUBJECT MATTER
IPC(6) :C03B 23/047,37/027
US CL :65/382, 321
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B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
U.S. : 65/382, 321

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 4-231,336 A (SETO et al) 20 August 1992, English abstract.	1-20

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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(71) Applicant: FIBERCORE, INC. [US/US]; 253 Worcester Road, P.O. Box 180, Charlton, MA 01507 (US).

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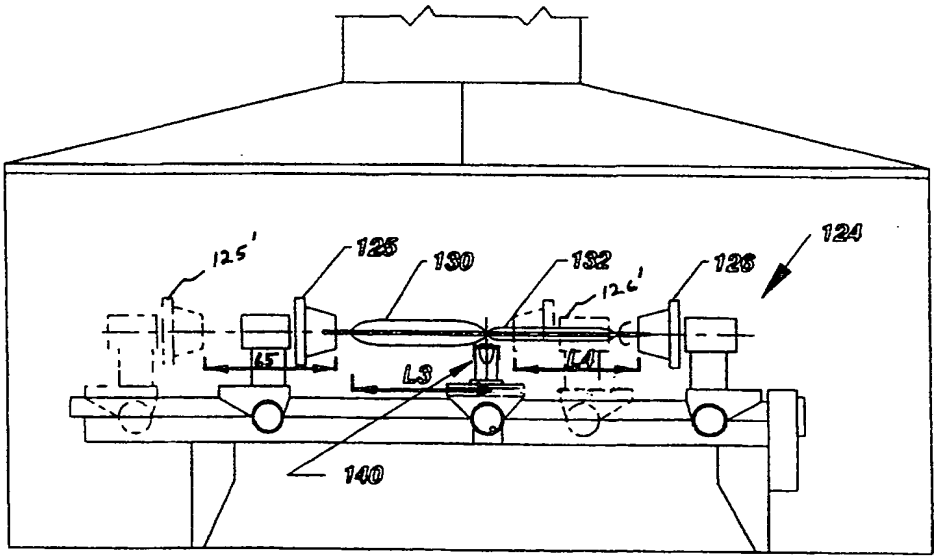
Published
With international search report.
With amended claims.

(54) Title: METHOD OF MAKING AN OPTICAL FIBER PREFORM



(57) Abstract

Glass soot (130) is deposited on a glass rod by a burner (140). The body is stretched (126). More depositing and stretching is effected. The final body is then drawn into a fiber.

*(Referred to in PCT Gazette No. 52/1999. Section II)

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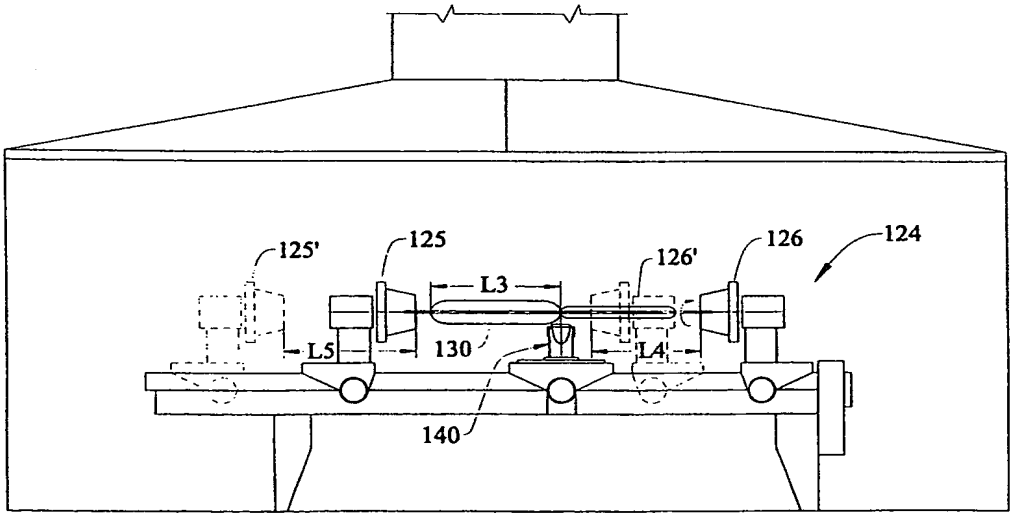

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METHOD OF MAKING AN OPTICAL FIBER PREFORM

BACKGROUND OF THE INVENTION

The present invention relates to methods for making optical fiber preform of both single mode and multimode design using a plasma outside vapor deposition process.

The prior art teaches various approaches for fabricating silica glass starter tubes, and for making optical fiber preforms. Starter tubes can be formed by heating silica and extruding it through an aperture. Both starter tubes and optical fiber preforms can be made by depositing doped or undoped silica onto a target using one of several techniques such as modified chemical vapor deposition (MCVD), vapor axial deposition (VAD), outside vapor deposition (OVD). Each of these methods starts with providing a rotating target, typically shaped in the form of a tube or a solid rod, and formed from glass, ceramic or one of several other materials. In certain cases, the rod or tube becomes an integral part of the preform but, in other cases, the rod will be removed. A heat source, such as a gas burner or a plasma source is positioned beneath, above or laterally, across the rotating target. The heat source will provide the required energy for the glass-forming reactions to form glass particles. Depending upon the nature of the process, these deposited glass particles are ready for the next processing, drying and sintering steps such as VAD or OVD processes. If it is an MCVD process, these particles will be fused into vitreous quartz by the same heat source.

When the target is mounted horizontally, the heat source travels along the length of the target to ensure uniform deposition. If the target is a tube, the glass forming particles and materials may be deposited either on the inside surface of the tube, in which case the

outer diameter remains constant, or on the outside of the tube, in which case the outer diameter grows.

When the target is mounted vertically, it rotates around its vertical axis, and with burners located either vertically above or laterally across, grows in both radial and axial directions. This results in a substantially cylindrical product whose diameter and length increase as deposition continues.

UPS 3,737,292 to Keck et al. discloses a method of forming optical fibers. Multiple layers with predetermined index of refraction are formed by flame hydrolysis and deposited on the outside wall of a starting rod or member. After these layers of glass are coated on the rod the resulting hollow cylinder is heated and collapsed to form fibers.

USP 4,224,046 to Izawa et al. teaches a method for manufacturing an optical fiber preform. Two gaseous glass forming materials, oxygen, hydrogen and argon are jetted upwards in a burner towards a vertically mounted, rotating cylindrical start member. Soot-like glass particles are formed by flame hydrolysis and deposited on the lower end of the start member. The start member is gradually withdrawn upwards to maintain a constant spacing between the its growing end and the burner. Upon completion of the deposition, the resulting soot-like glass preform is then dried and sintered to form a transparent glass preform.

UPS 4,217,027 to MacChesney et al. teaches the fabrication of preforms by what is usually referred to as the Modified Chemical Vapor Deposition (MCVD) process. In this process, a vapor stream consisting of chlorides or hydrides of silicon and germanium with oxygen is directed to the inside of a glass tube. The chemical reactions among these chemicals, which are preferentially induced by a traversing hot zone, will under proper conditions result in the formation of glass on the inner wall of the tube. The particular matter deposited on the tube is fused with each passage of the hot zone.

USP 4,412,853 to Partus discloses an MCVD process to form an optical fiber preform starter tube. The process starts with a horizontally mounted, rotating tubular target formed from glass and having a preselected composition and optical characteristics. A vapor stream is fed through the tubular target as a heat source positioned beneath the tubular target, traverses along the latter's length. This causes reaction products of the vapor stream to be deposited on, and fuse to, the interior surface of the tubular target. The deposited material has the same index of refraction as the tubular target, but a different composition. This reference also suggests that one may achieve the same effect by an outside vapor-phase oxidation process or an outside vapor-phase axial deposition process, but does not explicitly teach how this can be done.

USP 4,741,747 to Geittner et al. is directed to the Plasma Chemical Vapor Deposition (PCVD) method of fabricating optical fibers. In this PCVD method glass layers are deposited on the inner wall of a glass tube by heating the tube to a temperature between 1100° and 1300° C, before passing the reactive gas mixture at a pressure between 1 and 30 hPa, and moving a plasma back and forth inside the glass tube. After the glass layers are deposited, this glass tube is collapsed to produce a solid preform. Optical fibers can be drawn from this preform.

USP 5,522,007 to Drouart et al. teaches the use of plasma deposition to build up an optical fiber preform having high hydroxyl ion concentration. In this reference, hydroxyl ions are deliberately entrained in a plasma generating gas by passing the gas through a water tank before it is introduced into one end of a plasma torch having an induction coil. The plasma torch projects molten silica particles mixed with hydroxyl ions onto a rotating substrate preform. This results in a preform having an average hydroxyl ion concentration lying in the range to 50-100 ppm deposited onto the

target preform. According to Drouart et al., this technique results in optical fibers having an attenuation of 0.32 dB/km and 0.195 db/km at 1310 nm and 1550 nm, respectively.

In addition to requiring multiple processing steps to fabricate preforms, some other disadvantages of the above processes are that:

1. the MCVD and PCVD processes are slower processes because of their low deposition rate;

2. the preform size is limited by the size of the deposition tube for MCVD and PCVD process; and

3. the OVD and VAD processes are based on flame hydrolysis which generates excessive amounts of water and requires additional drying and sintering steps to produce high quality optical fiber preforms.

SUMMARY OF THE INVENTION

It is an object of the invention to provide a method for producing an optical fiber preform having low hydroxyl content at low cost by reducing the number of steps entailed in its manufacture, while increasing the size of a preform and increasing the rate of deposition. This and other objects are achieved by the present inventive method for forming an optical fiber preform.

In one aspect of the present invention, a plasma source is placed in proximity to a starter rod formed from a primary material. The starter rod is held horizontally at both ends and is arranged to rotate about its longitudinal axis. The plasma source is used to deposit silica doped with a known first doping concentration. The doped silica is deposited along the length of the starter rod until the latter grows to a desired diameter. The complex comprising the starter rod and the doped silica is then drawn down and a thinned section is extracted for use as a secondary rod. The secondary rod has a center formed from the primary material, and an outer layer formed from the doped silica. Additional silica, having the same doping

concentration, is deposited atop this secondary rod until it, too, reaches a desired diameter, and then is drawn down and a section extracted. The steps of depositing drawing down, extracting and depositing may be repeated a number of times. The result of this activity is a doped silica rod having a center formed from the primary material with a first diameter, and an annular layer formed from the doped silica with a second outer diameter.

The doped silica rod is subject to further processing. Specifically, the plasma source is used to deposit an outer layer of doped silica atop the doped silica rod and the resulting structure may then be drawn down and a thinned section extracted, as before. The dopant used in forming the outer layer may be selected to either increase, or decrease, the index of refraction of the silica.

If the dopant concentration is varied as the outer layer is being deposited, the outer layer is a graded layer. In such case, typically, the dopant concentration is varied from a maximum, beginning concentration level when the outer layer is first being deposited, to a minimum, end concentration level when deposition of the outer layer is almost complete.

If the dopant concentration is not varied as the outer layer is being deposited, the outer layer is a stepped layer. In such case, typically, a second dopant concentration, different from the first dopant concentration, is used throughout the deposition of the outer layer.

In yet another aspect of the present invention, the complex comprising the doped silica rod and the outer layer is subjected to further processing. The plasma source is used to deposit a cladding layer atop the outer layer. If the outer layer was graded, the cladding layer may be formed from silica doped with the same dopant and same minimum, end concentration level. Alternatively, the cladding layer can be formed from pure silica, or

even silica doped with some other dopant and at a third dopant concentration. If desired, the cladding layer may also have a graded doping.

In yet another aspect of the present invention, the complex comprising the doped silica rod, the outer layer and the cladding layer, is provided with a jacket. The jacket can be added by either further plasma deposition, or, alternatively, by providing a jacketing material over this complex and then applying heat to collapse the jacketing material into a finished preform.

During plasma deposition, a dry plasma gas having a low hydroxyl concentration is used to form the plasma. A dry quartz source gas comprising $SiCl_4$, or other similar source gases having low hydroxyl concentration, and a dopant source gas such as $GeCl_4$, which is sometimes co-doped with $POCl_3$ or PCl_5 are introduced in proximity to the plasma. This causes the material to be converted to silica (SiO_2), or silica doped with germanium oxide (GeO_2) and or phosphorous pentoxide (P_2O_5) and deposited onto the target and fused into vitreous quartz in one simple step.

BRIEF DESCRIPTION OF THE DRAWINGS

These and other features, aspects, and advantages of the present invention can be seen in the drawings in which:

Fig. 1 shows an apparatus used to perform plasma deposition;

Fig. 2 shows a partial side view of a plasmatron used in the apparatus of Fig. 1;

Fig. 3 shows a top view of a plasmatron similar to that shown in Fig. 2;

Fig. 4 shows a flow pattern of the plasma within the plasmatron of Fig. 3;

Fig. 5 shows an optical fiber preform made in accordance with the method of the present invention.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Fig. 1 shows an apparatus 20 used for plasma outside vapor deposition. The apparatus comprises a chamber 22 which is sealed so as to prevent impurities from being introduced into the final product.

Within the chamber 22 is a lathe 24, such as that available from Heathway Ltd. or Litton Engineering Lab. The lathe 24 has a headstock 25 and a tailstock 26. The headstock 25 and the tailstock 26 are provided with a pair of opposing rotating spindle chucks 28 which hold the ends of an elongated target 30 having a substantially cylindrical outer wall. The spindle chucks 28 rotate target 30, as indicated by arrow A1. A movable carriage 32 movably mounted to the lathe 24 is arranged to travel in either direction along the target, as indicated by double headed arrow A2.

A plasma source, shown generally as 40, is supported by carriage 32. Carriage 32 thus moves plasma source 40 along the length of the target 30. This results in the deposition of material on top of the target 30 to form an optical fiber preform. The spindle chucks 28 rotate the target 30 to ensure that material is uniformly deposited by the plasma source 40 around the target so as to form a tubular member 34 having nearly perfectly cylindrical outer walls.

In the preferred embodiment, the plasma source 40 positioned on the carriage 32 moves in both directions along a substantial portion of the length of the target 30. This allows the plasma source 40 to travel along this portion of the target 30 and deposit materials therealong.

Instead of moving the plasma source 40 along the length of the target, the target 30 may be moved while the plasma source 40 remains stationary. This can be realized by having the headstock 25 and the tailstock 26 of the lathe move the target in a reciprocating fashion so that all relevant portions of the target are brought directly above the plasma source 40.

As another alternative, a plurality of plasma sources may be spaced apart along the length of the target. This allows for reduced movement of either the headstock 25 and tailstock 26 of the lathe 24, or the carriage 32 to which the plasma sources are attached, depending on which of the two is configured to move. In the extreme case where a great number of plasma sources are provided all along the length of the target, no movement of either the carriage 32 or the headstock 25 and tailstock 26 of the lathe 24 is needed.

In the preferred embodiment, the plasma source 40 is a plasmatron torch having a dry plasma gas introduced into it through a first gas line 42 and a source gas introduced into it through a second gas line 44.

The plasma gas is substantially comprised of nitrogen and oxygen in an appropriate, predetermined proportion. Air may serve as the plasma gas. In such case, filtered air first passes through a first dryer 46 to remove moisture before entering the first gas line 42. This ensures that the hydroxyl concentration of the plasma gas is low, on the order of 2.0 ppm, or less. The total volume of gas being delivered will be regulated by a mass flow controller (MFC) 80 or by a flowmeter, as an alternative.

The source gas comprises a source chemical such as $SiCl_4$, and at least one carrier gas, such as oxygen O_2 or nitrogen N_2. The carrier gases enter the second dryer 48 to remove moisture. This ensures that the hydroxyl concentration of the source gas is also very low, on the order of 0.5 ppm. After the carrier gases are dried, they proceed to a MFC 81 before entering a bubbler 50 to pick up the source chemical. Depending upon the characteristics of the MFC, it is also possible to use it downstream of the bubbler. The gas stream comprising carrier gases laden with the source chemical then proceeds to the second gas line 44. Optionally, by opening valve 52, a dopant gas may be introduced into the gas stream before it reaches the plasmatron torch.

In the preferred embodiment, the source chemical is $SiCl_4$. This chemical is chosen for its reactive properties in a plasma. Specifically, the $SiCl_4$ serves as a source of Si to form SiO_2 which is deposited on the target 30. The dopant can be a fluorine dopant gas in the form of SiF_4 or SiF_6. Fluorine dopants will lower the index of refraction and also change the viscosity of the quartz. In addition, fluorine dopants result in increased design flexibility for optical fiber preforms. As is well known, however, if one wishes to increase the index of refraction, GeO_2 or other equivalent substance may be used as the dopant.

In the preferred embodiment, the source chemical for GeO_2 is $GeCl_4$. This chemical is chosen for its purity because of its having similar physical and chemical properties $SiCl_4$. The delivery of the $GeCl_4$ will be similar to $SiCl_4$. The carrier gas from the dryer 48, can be split to another branch where it will be regulated by a MFC 82, before proceeding to a bubbler 83 to pick up the source chemical $GeCl_4$. Similar to the control of chemical $SiCl_4$, the MFC can also be located downstream of the bubbler. This gas stream can feed into the gas line 44 and form a mixture before entering the plasmatron torch. It is also possible to directly introduce the $GeCl_4$ gas stream by a separate line 84 to the plasmatron torch. One advantage of using the separated delivery lines is to minimize the competing chemical reactions between $GeCl_4$ and $SiCl_4$. Other source chemicals that can be used for doping instead of germanium oxide (GeO_2) or co-doping with germanium oxide are materials such as $POCl_3$, PCl_5, and other similar index increasing dopants such as Aluminum and Titanium containing chemicals.

Fig. 2 shows a cutaway side view of the plasmatron torch 40 positioned below the target 30. The plasmatron torch 40 comprises a substantially tubular torch housing 50 formed from quartz. The housing has a diameter of 60 mm and a height of 220 mm. However, diameters ranging

from 40-80 mm and heights between 180-400 mm may also be used.

A copper induction coil 52 is provided around the upper portion of the housing 50. The coil 52 comprises a plurality of windings 54 having a diameter of approximately 72 mm and spaced apart from each other by 6 mm. A gap between the housing and the coil can be between 2-10 mm. The uppermost portion of the coil 52, as indicated by uppermost winding 54', is separated from the outer surface of the tubular member 34 by a spacing designated by L, which is on the order of 30-55 mm.

As the quartz glass is deposited, its outer diameter increases. However, the spacing L is maintained by adjusting the height of a support stand 56 on which the plasma torch 40 is placed. Support stand 56, in turn, is mounted to carriage 32, and moves laterally therewith. Initially, the support stand 56 is set at a predetermined height, and this height is reduced as the diameter of the deposited material increases during deposition. This maintains a predetermined distance between the plasma torch 40 and the deposited material. An optical or other sensor mounted on the carriage 32 and connected to a controller may be used to gauge the distance of the radially growing tubular member 34 from the carriage, and adjust the height of the support stand 56, accordingly.

On either side of the uppermost portion of the housing 50 is a plasma stabilizer bar 58. Each stabilizer bar is formed from quartz and comprises a U-shaped gutter extending laterally from the rim of the housing 50. The stabilizer bars 58 have a diameter of 60 mm and extend 20 mm on diametrically opposite sides of the housing rim, although diameters in the range of 40-80 mm and lengths of 15-40 mm may also be used. When the plasmatron torch 40 is in use, the stabilizer bars 58 are aligned parallely to the target. This arrangement helps spread the reactive source chemicals being deposited onto the growing tubular member 34.

A pair of injection ports 60 connect the second gas line 44 carrying the source chemicals to the plasmatron torch 40. The injection ports 60 enter the housing at substantially the same height along the housing 50, at a point between the uppermost windings 54' of the coil 52 and the stabilizer bars 58. The injection ports comprise quartz tubing having a diameter of 5 mm, although tubing diameters on the order of 3-10 mm may be used with the plasmatron torch 40 of the present invention. In the preferred embodiment, a pair of injection ports 60 enter the housing 50 at the same height and are positioned diametrically across from each other. Instead of just two such ports, however, three or even more ports, symmetrically arranged, may be provided. In Fig. 2, the two injection ports 60 are shown to be directly beneath the stabilizer bars. This, however, is not an absolute necessity, and the injection ports 60 may be angularly offset from the stabilizer bars 58, in a top view of the plasmatron torch, as shown in Fig. 3.

A pair of plasma gas inlets 62 connect the first gas line 42 carrying the plasma gases to the plasmatron torch 40. The plasma gas inlets 62 enter the housing at substantially the same height, proximate to the base of the housing. These inlets 62 comprise stainless steel tubing having a diameter of 5 mm, although a range of diameters may suffice for this purpose.

The plasmatron torch 40 is also provided with a coolant inlet 64 and outlet 66. During use, a coolant, such as water, passes through the inlet 64, circulates within the outer wall of the housing 50, and exits through the outlet 66. The coolant inlet and outlet are formed from stainless steel and have a diameter of 5 mm. As with the plasma gas inlet and the injection port, this diameter may also vary.

The plasma gas inlets 62, the coolant inlet 64 and the coolant outlet 66 are all formed in a stainless steel chamber 68. The chamber 68 is a stainless steel square block 80 mm on a side, and having a height of

approximately 40 mm. The chamber 68 is mounted onto the support stand 56 which, in turn, is mounted on the carriage 32 for movement along the target 30.

A high frequency generator (not shown) is electrically connected to the coil 52, powering the latter with a variable power output up to 80 kW at a frequency of approximately 5.0 MHz. In the preferred embodiment, the generator is Model No. T-80-3MC from Lepel Corporation. This generator is driven with a 60 Hz, 3-phase 460 V power supply to energize the plasmatron torch 40. As an alternative, a Model No. IG 60/5000 generator is available from Fritz Huttinger Electronic GmbH of Germany.

Fig. 4 depicts the plasma jet 70 formed within the plasmatron torch 40 when the dry plasma gas is fed through the inlets 62 and converted into a plasma. The plasma jet 70 is substantially symmetric about the torch's longitudinal axis A'. The position of the injection ports 60 is such that the source chemicals are introduced into the plasma just above a point V where the vertical velocity of said plasma is zero. This provides the needed structure of hydrodynamic and thermal flow of the source chemical jet into the border layers to realize efficient deposition onto the growing tubular member 34. And while the preferred embodiment has the injection ports entering laterally into the housing, this is not an absolute requirement. Instead, the source gases may introduced into the center of the plasma jet 70 by a water cooled probe extending along the longitudinal axis A' of the plasmatron torch 40.

Fig. 5 illustrates a well-known procedure which can performed with a lathe 124, such as Model No. PFH842XXLS Precision Quartz and Glass Working Lathe, manufactured by Heathway. The headstock 125 and tailstock 126 of the lathe 124 can move longitudinally relative to one another. This allows for easy loading and unloading of a finished workpiece 130 of length L3 which has been deposited atop an initial target. More significantly, it

also allows one to draw down a portion of a workpiece into a secondary rod of a reduced diameter comparable to that of the original target. This is accomplished by keeping the headstock 125 stationary and moving the tailstock 126 away from the headstock 125 while the plasma source 140 is moved in a direction opposite to that of tailstock 126. Alternatively, this can also be accomplished by placing a plasma source 140, or other heat source, at one end of the workpiece 130 to soften it. Then the headstock 125 and tailstock 126 are moved in the same direction, but with different speeds by distances L5, L4, respectively, to the positions shown in phantom 125', 126'. The result is a thin, secondary rod 132, which can (but need not) have the same diameter as the original target. As is known to those skilled in the art, the secondary rod has the same cross-sectional composition as the workpiece from which it is derived, as so has a center whose consistency along is substantially similar to that of the original target, and outer layer substantially similar to the materials deposited atop the target during the formation of the workpiece.

The lathe 124 allows the headstock 125 and tailstock 126 to be moved far enough longitudinally to stretch the secondary rod to a distance L4, which is substantially the same as the length L3 of the workpiece from which it is derived. The secondary rod 132 may be cut from the workpiece, mounted on the lathe 124 in place of the workpiece 130, and used as a target for subsequent deposition with the plasma source 140. Thus, the original, or first-generation, target is used to create a first-generation workpiece, from which a secondary rod can be drawn to be used as a second-generation target. Deposition atop this second-generation target can will thereby form a second-generation workpiece, and so on. This iterative process of plasma deposition on a target to form a workpiece, stretching one end of the workpiece to form a reduced-diameter rod, and using this reduced-

diameter rod as a subsequent target for further deposition can be repeated an arbitrary number of times.

If the material being deposited atop the target is unchanged through the iterations, the result of N iterative steps is an N-th generation rod having a very small center which is substantially identical in composition to the original target, and an annular layer reflective of the materials deposited atop the target. For instance, if the original target has a diameter D1 and the finished workpiece has a diameter D2 = M x D1, then the proportion of the original target material in the first-generation workpiece is approximately $1/M^2$. If a second-generation target of diameter D1 is drawn from this workpiece and material sufficient to form a second-generation workpiece of diameter D2 is deposited thereon, the proportion of the original target material in the second generation workpiece is approximately $1/M^4$. Thus, it can be seen that one may readily form a workpiece having a predetermined proportion of the original target material therein by controlling M during deposition, along with the total number of iterations.

A method for forming a multimode optical fiber preform using the aforementioned iterative technique will now be described. In order to provide a more detailed explanation, some dimensions are given. However, it must be noted that in the actual process, many different value are possible.

The method begins by providing a first generation target, horizontally mounted on a lathe, such as that shown in Fig. 5. The target is preferably formed from pure silica, in which case it may be purchased from a commercial vendor, such as Product no. F300, available from Heraeus Amersil of Georgia. Alternatively, the first-generation target may be an Nth-generation doped silica rod formed using the current process. In the preferred embodiment, the first-generation target has a length of one meter and a diameter D1 = 6 mm.

Silica doped with GeO_2 is deposited atop the first-generation target using the plasma source described above. The dopant concentration for the GeO_2 depends on the desired numerical aperture (NA) of the multimode optical fiber being produced. For instance, to form a fiber with a NA of 0.2, the maximum GeO_2 dopant concentration is approximately 10%. And to form a fiber with a NA of 0.275, the maximum GeO_2 dopant concentration will be approximately 18%.

The dopant concentration may be held at the same level during deposition, in which case a stepped layer, is formed. Alternatively, the dopant concentration may be gradually varied to form a graded layer. This is done by automatically controlling, by means of a microprocessor or like, an adjustable flow meter through which the dopant is introduced. It should be noted that stepped and graded layers may succeed one another in subsequent generations of workpieces, and that layers having different, constant doping concentrations may succeed one another, as well. Thus, a graded layer may be deposited on the first-generation target, and a stepped layer may be deposited atop the second-generation target formed after drawing down the first-generation workpiece. Similarly, one may deposit a stepped layer atop a graded layer, which has been deposited atop an original first-generation target. Also, a first stepped layer, having a first dopant concentration, may be deposited atop a target, and a second stepped layer, having a second dopant concentration, deposited atop the next generation target. Additional layers, either graded or stepped, may be deposited atop any of the above structure.

In the preferred embodiment, silica doped with 18 % GeO_2 is deposited as a stepped layer atop the 6 mm diameter first generation target until a workpiece having a length of one meter and a diameter of D2 = 48 mm is formed (i.e., M = 8). This resulting first-generation workpiece has approximately 64 times the cross-sectional

area of the original first-generation target. The first-generation workpiece is then drawn down into 64 first-generation doped silica rods, each having a length of one meter and a diameter of 6 mm. Each of these doped silica rods may then be used as a second-generation target.

The second generation target is placed in a lathe and a second deposition layer is applied to form a second-generation workpiece having a 48 mm diameter. This second deposition is carried out with the same, constant dopant concentration as the first deposition. Maintaining the dopant concentration at the same level throughout the deposition process results in a first-generation doped silica rod with a center formed from the original target material and an annular layer which has substantially the same composition therethrough. This ensures that the optical properties of the second layer is substantially the same as that of the first layer which was deposited on the original target. The second-generation workpiece is then drawn down into 144 second-generation doped silica rods, each having a length of one meter and a diameter of 4 mm. Each of these may be used as a third-generation target. It should be noted here that the iterative process may continue with the deposition of additional layers having the same dopant concentration. At some point, however, a workpiece with a desired proportion of original target material will be formed, after which no further iterations are needed. Indeed, this may even be reached after the first generation workpiece is formed.

In the preferred embodiment, a graded deposition layer having an outer diameter of approximately 80 mm is deposited atop the 4 mm diameter third-generation target. The dopant concentration starts out at a maximum value of 18% GeO_2 closest to the outer surface of the third-generation target, and is gradually reduced to a minimum value approximately 0.1% GeO_2 at its outermost portion, where the diameter is about 80 mm. This results in a third-generation workpiece having a center formed from

the original target, two layers having substantially the same optical properties and fairly indistinguishable from one another, and a third, graded layer.

In the preferred embodiment, the 80 mm diameter third-generation workpiece is subject to additional processing to form a primary optical fiber preform. Specifically, a cladding, or barrier, layer is deposited atop the third-generation workpiece. The thickness of the cladding layer depends on the type of finished optical fiber preform to be made. For a 62.5/125 fiber preform, the finished primary preform will have a final diameter of about 93 mm. For a 50/125 fiber preform, the finished primary preform will have a final diameter of about 96 mm. The cladding layer is formed by depositing silica doped at the same concentration of GeO_2 as the minimum doping concentration level used to form the third layer, i.e., 10% GeO_2. This results in a structure having the original target material at the center, a constantly doped pair of second layers having the same optical properties, a graded layer having a dopant concentration varying from a maximum value to a minimum value, and a cladding layer comprising silica doped at the minimum value.

Once the cladding layer is applied, the finished primary preform must be stretched to form the final preforms. From a single, 1 meter long 62.5/125 preform having a diameter of 93 mm diameter, one can obtain eight, one-meter long preform pieces, each having an outer diameter of 32 mm. And from a single, 1 meter long 50/125 preform having a diameter of 96 mm diameter, one can obtain twelve, one-meter long pieces, each having an outer diameter of 27 mm.

A jacketing layer may be applied atop the cladding layer of these preform pieces. The jacketing layer preferably has the same index of refraction as pure silica. The jacket may be applied by plasma outside vapor deposition using pure silica. Alternatively, a tube or sheet of pure silica, having an appropriate

diameter or width, may be provided around a preform piece, and heat applied to fuse the jacket onto the preform piece to form the final optical fiber preform. In the preferred embodiment, the final optical preform has an outer diameter of about 56 mm. This final preform may then be drawn into approximately 200 Km of fiber having a diameter of 125 μm.

Although, for best performance, a cladding and then a jacketing layer is applied, it should be noted that one may dispose of the cladding step and directly apply a jacketing tube to the third-generation workpiece, once it has been stretched.

A similar method for making single mode optical fiber preform can be achieved by using the following procedure. The starting target can be a pure silica rod that can be either a F300 rod purchased from Heraeus or a pure silica Nth-generation rod fabricated in house. Multiple fluorine doped silica layers with constant concentration are deposited on the target until it reaches a desired diameter. Single mode optical fibers can be drawn from this preform. There are many different glass index modifiers such as F, GeO_2, P_2O_5, TiO_2, Al_2O_3, etc., and in the proper combination, they can be used to make the doped core and/or doped cladding. In the preferred embodiment, the target is a Nth-generation GeO_2 doped rod with pure silica or doped silica cladding layers deposited on it. The preform is completed when the desired diameter is reached.

While the present invention has been disclosed with reference to certain preferred embodiments, these should not be considered to limit the present invention. One skilled in the art will readily recognize that variations of these embodiments are possible, each falling within the scope of the invention, as set forth in the claims below.

CLAIMS

What is claimed is:

1. A method for making an optical fiber preform comprising the steps of:

(a) providing a target rod formed from a first material;

(b) depositing atop said target rod, a first silica layer doped with a first dopant provided at a first concentration, said first silica layer being deposited to a predetermined first thickness;

(c) drawing down said target rod with said first silica layer deposited thereon to a predetermined first diameter, thereby forming a doped silica rod;

(d) repeating steps (b) and (c)

(d1) for a predetermined number of times, or

(d2) until said first material comprises a predetermined proportion of said doped silica rod;

(e) depositing atop said doped silica rod, a second layer comprising silica doped with a second dopant provided at a second concentration, said second silica layer being deposited to a predetermined second thickness to thereby form an intermediate structure;

(f) depositing a third layer atop said intermediate structure, said third layer being deposited to a predetermined third thickness to thereby form a preform structure.

2. The method of claim 1, comprising the additional step of:

(g) applying a jacketing layer atop said preform structure, said jacketing layer consisting essentially of pure silica and being applied to a predetermined fourth thickness.

3. The method of claim 2, comprising the additional step of:

drawing down said preform structure to a predetermined third diameter after step (f) and prior to step (g).

4. The method of claim 1, wherein the first material is one from the group consisting of silica and silica doped with a dopant.

5. The method of claim 4, wherein the dopant is an index modifying material which is one from the group consisting of F, GeO_2, P_2O5, TiO_2 and Al_2O_3.

6. The method of claim 1, wherein, in step (e), said second concentration differs from said first concentration, said method comprises the additional step of:

maintaining the second concentration at a constant value as the second silica layer is being deposited, thereby forming a step index profile in the indices of the refraction of the doped silica rod and the second silica layer.

7. The method of claim 1, comprising the step of varying the second concentration as the second silica layer is deposited.

8. The method of claim 7, wherein
the second dopant is fluorine, and
said second concentration is varied from a minimum value when said second silica layer is first being deposited, to a maximum value when deposition of said second silica layer is nearing completion.

9. The method of claim 7, wherein said second concentration is varied from a maximum value when said second silica layer is first being deposited, to a minimum value when deposition of said second silica is nearing completion.

10. The method of claim 9, wherein said maximum value of said second concentration is substantially the same as said first concentration.

11. The method of claim 9, wherein the third layer deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with said second dopant at said minimum value.

12. The method of claim 9, wherein the third layer deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with fluorine.

13. The method of claim 1, wherein the third layer deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with fluorine.

14. The method of claim 9, comprising the additional step of:

(g) applying a jacketing layer atop said preform structure, said jacketing layer consisting essentially of pure silica and being applied to a predetermined fourth thickness.

15. The method of claim 14, comprising the additional step of:

drawing down said preform structure to a predetermined third diameter after step (f) and prior to step (g).

16. The method of claim 1, wherein at least one of said depositing steps (b), (e) and (f) is performed by plasma outside vapor deposition, which comprises the steps of:

providing a high-frequency plasmatron comprising a coil, said plasmatron being selectively positionable along a length of said target with a spacing of 30-55 mm separating the target from said coil;

introducing a plasma gas having a hydroxyl content of less than 2 ppm into the plasmatron to form a plasma;

injecting a source gas comprising at least $SiCl_4$ and a dopant into a region in communication with said plasma, said source gas having a hydroxyl content of less than 0.5 ppm; and

depositing at least one reaction product of said plasma and said source gas onto the target while maintaining said spacing between the target and the coil.

17. The method of claim 16, wherein the source gas is introduced just above a point in the plasmatron at which the vertical velocity of the plasma is zero.

18. The method of claim 16, wherein said coil comprises a plurality of windings, and the target is separated from a winding closest to the target by said spacing.

19. The method of claim 17, comprising the additional step of drying the plasma gas before it is introduced into the plasmatron.

20. The method of claim 1, wherein the first and second dopants and the first and second concentrations, are the same.

AMENDED CLAIMS

[received by the International Bureau on 10 August 1999 (10.08.99) ;
original claims 1, 6, 16, 17, 18, 19 and 20 amended ;
remaining claims unchanged (4 pages)]

1. A method for making an optical fiber preform comprising steps of:

(a) providing a target rod formed from a first material;

(b) concurrently depositing and sintering on said target rod, by plasma torch, a first silica layer doped with a first dopant provided at a first concentration, said first silica layer being deposited and sintered to a predetermined first thickness;

(c) drawing down said target rod with said first silica layer deposited thereon to a predetermined first diameter, thereby forming a doped silica rod;

(d) repeating steps (b) and (c)

(d1) for a predetermined number of times, or

(d2) until said first material comprises a predetermined proportion of said doped silica rod;

(e) depositing on said doped silica rod a second layer comprising silica doped with a second dopant provided at a second concentration, said second silica layer being deposited to a predetermined second thickness to thereby form an intermediate structure;

(f) depositing a third layer on said intermediate structure, said third layer being deposited to a predetermined third thickness to thereby form a preform structure.

2. The method of claim 1, comprising the additional step of:

drawing down said preform structure to a predetermined third diameter after step (f) and prior to step (g).

4. The method of claim 1, wherein the first material is one from the group consisting of silica and silica doped with a dopant.

5. The method of claim 4, wherein the dopant is an index modifying material which is one from the group consisting of F, GeO_2, P_2O5, TiO_2 and Al_2O_3.

6. The method of claim 1, wherein, in step (e), said second concentration differs from said first concentration, and said method comprises the additional step of:

maintaining the second concentration at a constant value as the second silica layer is being deposited, thereby forming a step index profile in the indices of the refraction of the doped silica rod and the second silica layer.

7. The method of claim 1, comprising the step of varying the second concentration as the second silica layer is deposited.

8. The method of claim 7, wherein the second dopant is fluorine, and said second concentration is varied from a minimum value when said second silica layer is first being deposited, to a maximum value when deposition of said second silica layer is nearing completion.

9. The method of claim 7, wherein said second concentration is varied from a maximum value when said second silica layer is first being, deposited, to a minimum value when deposition of said second silica is nearing completion.

10. The method of claim 9, wherein said maximum value of said second-concentration is substantially the same as said first concentration.

11. The method of claim 9, wherein the third layer deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with said second dopant at said minimum value.

12. The method of claim 9, wherein the third layer

deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with fluorine.

13. The method of claim 1, wherein the third layer deposited in step (f) is a cladding layer deposited by plasma outside vapor deposition, said cladding layer consisting essentially of silica doped with fluorine.

14. The method of claim 9, comprising the additional step of:

(g) applying a jacketing layer on said preform structure, said jacketing layer consisting essentially of pure silica and being applied to a predetermined fourth thickness.

15. The method of claim 14, comprising the additional step of:

drawing down said preform structure to a predetermined third diameter after step (f) and prior to step (g).

16. The method of claim 1, wherein at least one of said steps (b), (e) and (f) is performed by plasma outside vapor deposition, which comprises steps of:

providing a high-frequency plasma torch comprising a coil having a plurality of windings around a coil axis, said plasma torch being selectively positionable along a length of said target with a spacing of 30-55 mm separating the target from said coil;

introducing a plasma gas having a hydroxyl content of less than 2 ppm into the plasma torch to form a plasma;

injecting a source gas comprising at least $SiCl_4$ and a dopant into a region in communication with said plasma, said source gas having a hydroxyl content of less than 0.5 ppm; and

depositing at least one reaction product of said plasma and said source gas onto the target while maintaining said spacing between the target and the coil.

17. The method of claim 16, wherein the source gas is introduced just above a point in the plasma torch at which the velocity of the plasma in the direction of said coil axis is zero.

18. The method of claim 16, wherein the target is separated from a winding closest to the target by said spacing.

19. The method of claim 17, comprising the additional step of drying the plasma gas before it is introduced into the plasma torch.

20. The method of claim 1, wherein the first and second dopants and the first and second concentrations are the same.

FIG. 1

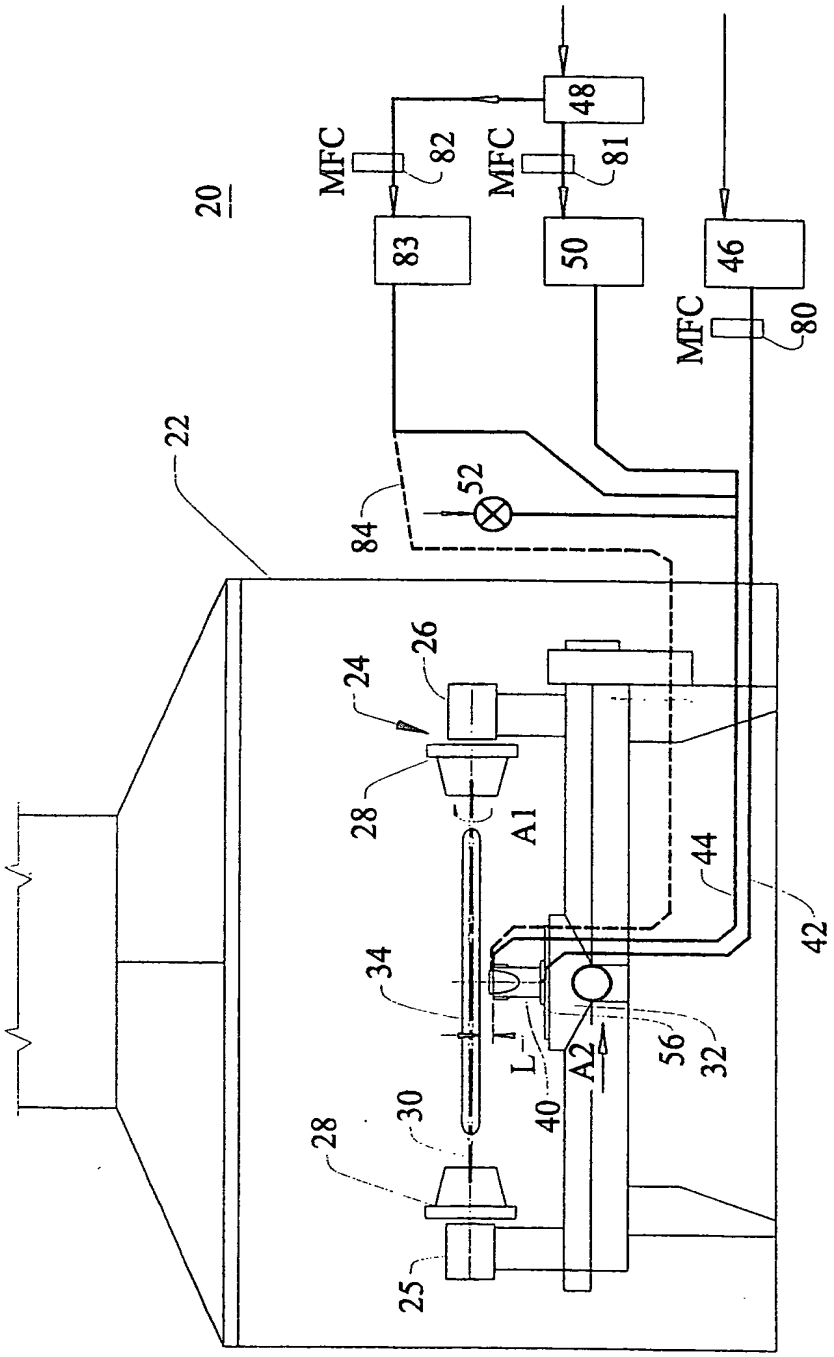


FIG. 2

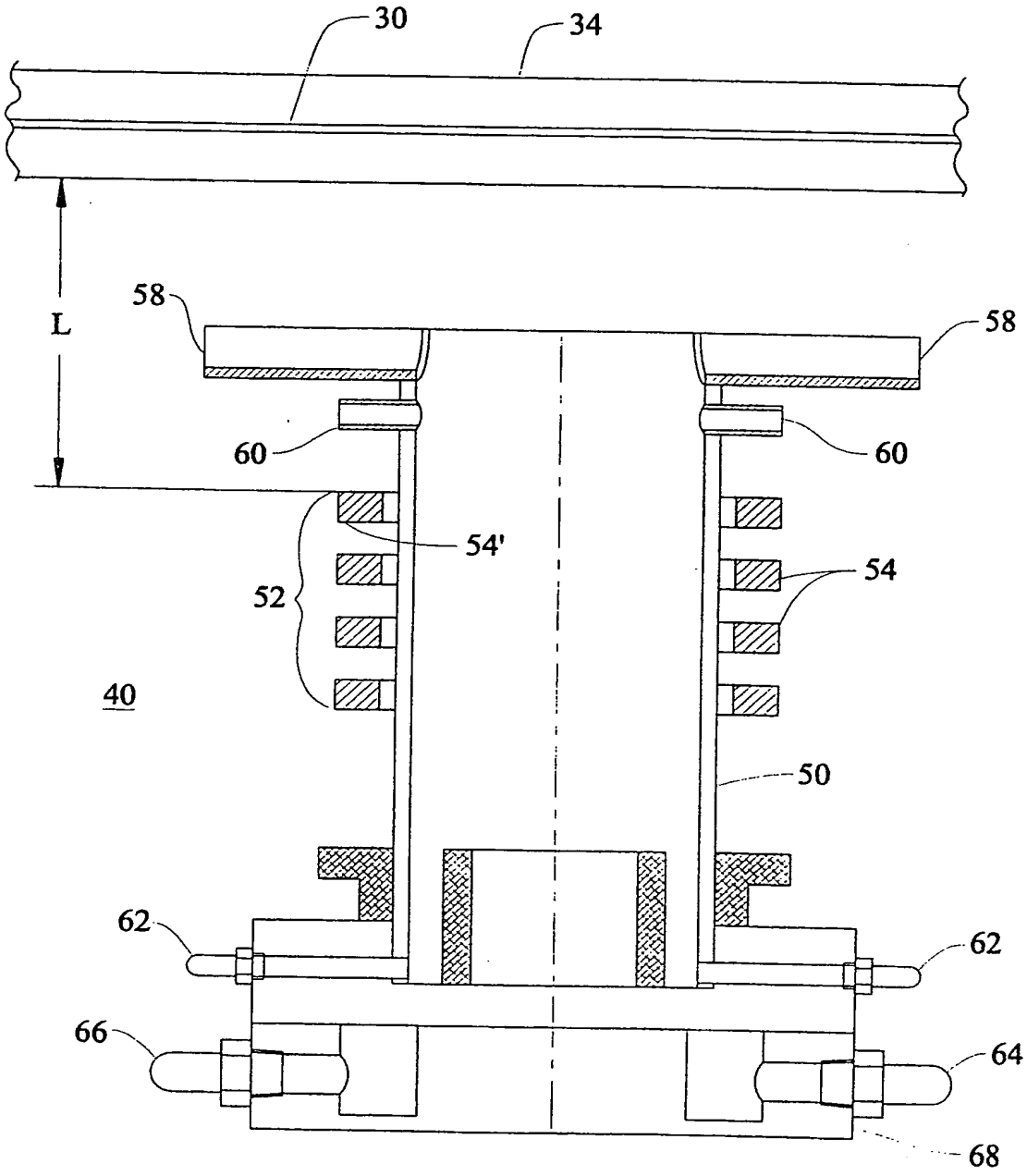


FIG. 3

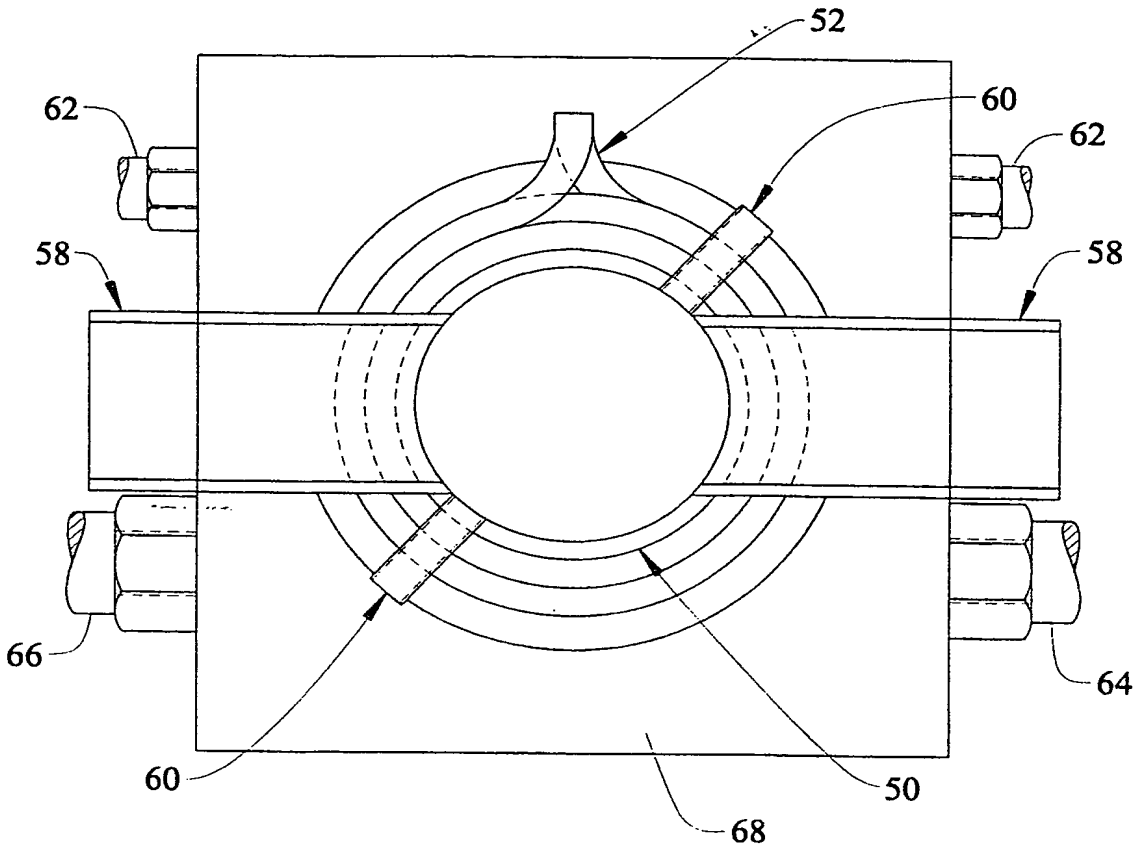


FIG. 4

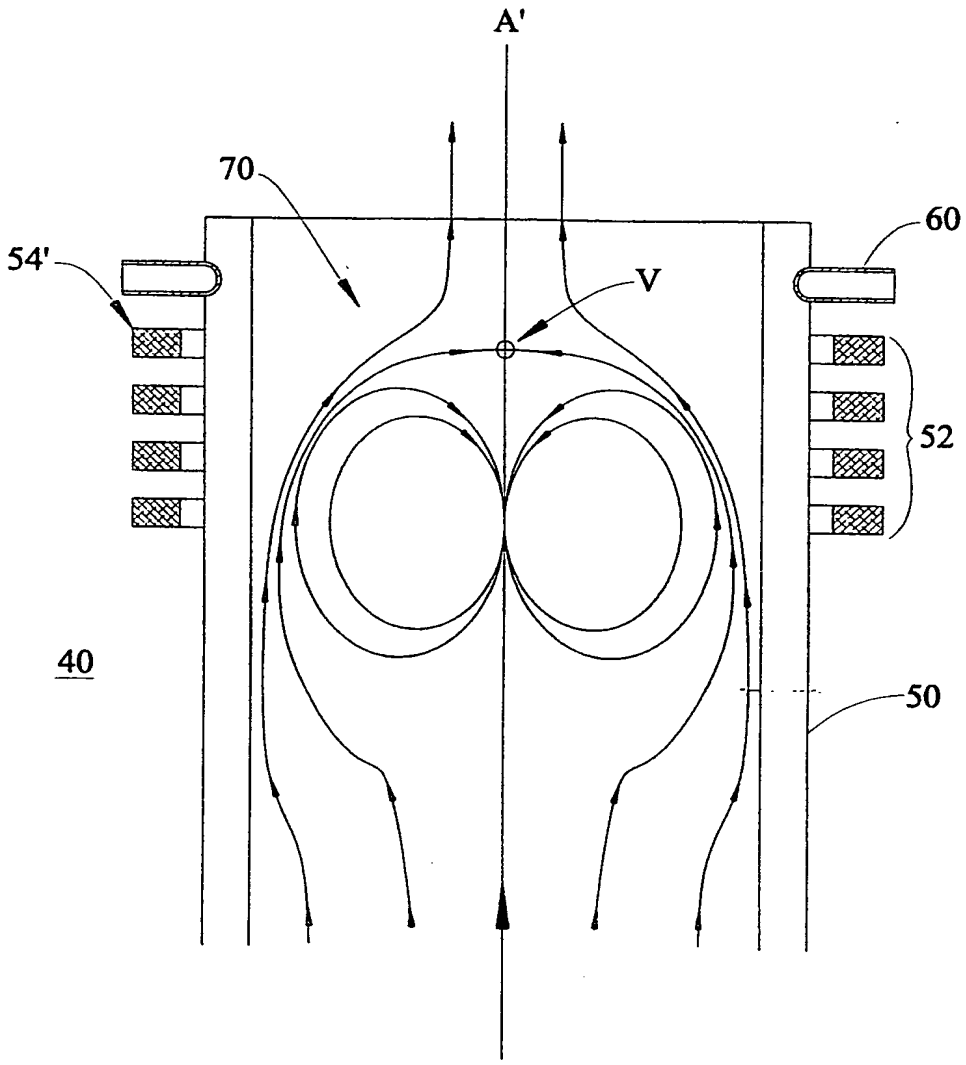
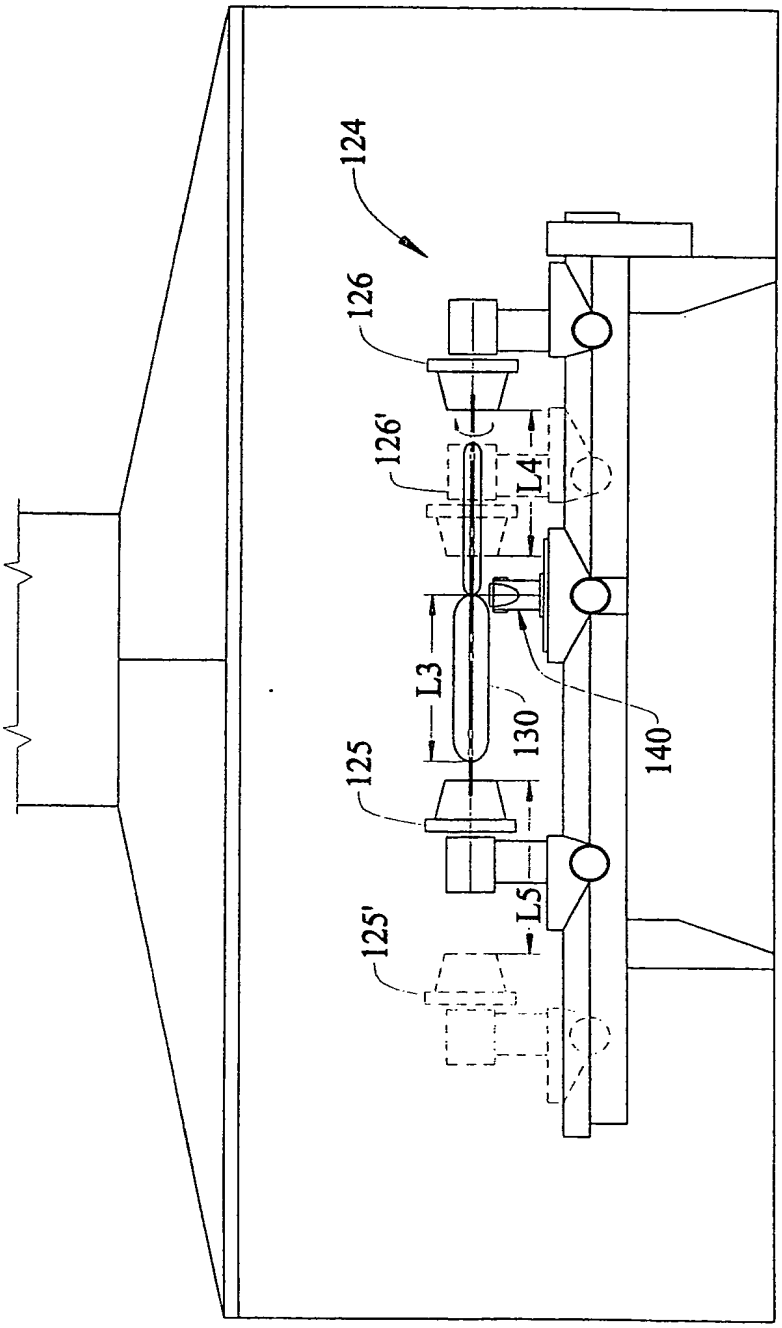


FIG. 5



INTERNATIONAL SEARCH REPORT

International application No.
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A. CLASSIFICATION F SUBJECT MATTER
IPC(6) :C03B 23/047,37/027
US CL :65/382, 321
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 65/382, 321

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	JP 4-231,336 A (SETO et al) 20 August 1992, English abstract.	1-20

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

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Date of the actual completion of the international search 14 MAY 1999	Date of mailing of the international search report 28 MAY 1999
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